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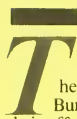
U.S. DEPARTMENT OF COMMERCE/National Bureau of Standards

*Standard Reference Materials:*

**Summary of the Coal,  
Ore, Mineral, Rock,  
and Refractory  
Standards Issued by the  
National Bureau of  
Standards**

**Radu Mavrodineanu and Thomas E. Gills**

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- Chemical Physics
- Analytical Chemistry

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<sup>2</sup>Some divisions within the center are located at Boulder, CO 80303.

# Summary of the Coal, Ore, Mineral, Rock, and Refractory Standards Issued by the National Bureau of Standards

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## Preface

Standard Reference Materials (SRM's) as defined by the National Bureau of Standards (NBS) are well-characterized materials, produced in quantity and certified for one or more physical or chemical properties. They are used to assure the accuracy and compatibility of measurements throughout the Nation. SRM's are widely used as primary standards in many diverse fields in science, industry, and technology, both within the United States and throughout the world. They are also used extensively in the fields of environmental and clinical analysis. In many applications, traceability of quality control and measurement processes to the national measurement system is carried out through the mechanism and use of SRM's. For many of the Nation's scientists and technologists it is therefore of more than passing interest to know the details of the measurements made at NBS in arriving at the certified values of the SRM's produced. An NBS series of papers, of which this publication is a member, called the NBS Special Publication - 260 Series, is reserved for this purpose.

This 260 Series is dedicated to the dissemination of information on different phases of the preparation, measurement, certification and use of NBS SRM's. In general, much more detail will be found in these papers than is generally allowed, or desirable, in scientific journal articles. This enables the user to assess the validity and accuracy of the measurement processes employed, to judge the statistical analysis, and to learn details of techniques and methods utilized for work entailing the greatest care and accuracy. These papers also should provide sufficient additional information not found on the certificate so that new applications in diverse fields not foreseen at the time the SRM was originally issued will be sought and found.

Inquiries concerning the technical content of this paper should be directed to the author(s). Other questions concerned with the availability, delivery, price, and so forth, will receive prompt attention from:

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Taylor, J. K., Standard Reference Materials: Handbook for SRM Users, NBS Spec. Publ. 260-100 (In Press).

\* Send order with remittance to Superintendent of Documents, US Government Printing Office Washington, DC 20402. Remittance from foreign countries should include an additional one-fourth of the purchase price for postage.

\*\* May be ordered from: National Technical Information Services (NTIS). Springfield Virginia 22161.

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#### Abstract

This publication is a summary of the coal, ore, mineral, rock, and refractory standards issued by NBS as Standard Reference Materials (SRM's). The material, composition, certification, use, and remarks concerning each of the SRM's described are presented in tabular form. Copies of the certificates of these SRM's are contained in the appendix for more detailed information.

Key Words: Chemical composition; coals; ores; refractories; rocks; Standard Reference Materials.

## Introduction

Since its inauguration in 1901, the National Bureau of Standards (NBS) has issued nearly 2000 different Standard Reference Materials (SRM's). Many of these have been renewed several times, many have been replaced or discontinued as technology changed. Today, over 900 SRM's are available, together with a large number of scientific publications related to the fundamental and applied characteristics of these materials. Each material is certified for chemical composition, chemical properties, or its physical or mechanical characteristics. Each SRM is provided with a Certificate or Certificate of Analysis that contains the essential data concerning its properties or characteristics. The SRM's currently available cover a wide range of chemical, physical, and mechanical properties, and a corresponding wide range of measurement interests in practically all aspects of fundamental and applied science. These SRM's constitute a unique and invaluable means of transferring to the user accurate data obtained at NBS, and provide essential tools that can be used to improve accuracy in practically all areas where measurements are performed.

In addition to SRM's, the National Bureau of Standards issues a variety of Research Materials (RM's) having various properties described in individual "Reports of Investigation." They are intended primarily to further the scientific or technical research on that particular material. Other materials, called Special Reference Materials (GM's), are also available from NBS. These are materials produced and certified by other Government agencies, standard organizations, or other nonprofit organizations, that are considered useful to the public and for which no alternate method of national distribution exists.

The various categories of materials available from NBS are given in Table 1. This table lists these materials according to their chemical composition, physical properties, or engineering characteristics. A more detailed alphabetic enumeration of these materials is given in Appendix I. Table 1 and Appendix I were taken from NBS Special Publication 260, NBS Standard Reference Materials Catalog, 1984-85 Edition<sup>1</sup>. This publication lists every material available from the NBS Office of Standard Reference Materials.

Further information on the reference materials available from NBS may be obtained from the Office of Standard Reference Materials, National Bureau of Standards, Gaithersburg, MD 20899. Information on other NBS services may be obtained from the Technical Information and Publications Division, National Bureau of Standards, Gaithersburg, MD 20899.

In addition to these types of materials, NBS provides many additional services. These include: Measurement Assurance Programs, Calibration and Related Measurement Services, Proficiency Sample Programs, a National Voluntary Laboratory Accreditation Program, Standards Information Services, Standard Reference Data, and Technical Information and Publications.

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<sup>1</sup>For sale by the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402, under Stock No. 003-003-02558-5 (Price \$5.50, add 25 percent for foreign orders.)



Table 1. Categories of Standard Reference Materials available from the National Bureau of Standards.

CERTIFIED CHEMICAL COMPOSITION STANDARDS

Steels (chip form)	Gases in Metals
Plain carbon	High-Purity Metals
Low alloy	Electron Probe Microanalytical Standards
High alloy	Primary, Working, and Secondary
Stainless	Standard Chemicals
Tool	Microchemical Standards
Steels (granular form)	Clinical Laboratory Standards
Steels (solid form)	Biological Standards
Ingot iron and low alloy	Environmental Standards
Special ingot irons and low alloy	Analyzed gases
Stainless	Analyzed liquids and solids
Specialty	Permeation tubes
High-temperature alloys	
Tool	
Steelmaking Alloys	Industrial Hygiene Standards
Cast Irons (chip form)	Forensic Standards
Cast Steels, White Cast Irons, Ductile	Hydrocarbon Blends
Irons, and Blast Furnace Irons	Metallo-Organic Compounds
(solid form)	Fertilizers
Nonferrous Alloys (chip form)	Ores
Aluminum "Benchmarks"	Minerals, Refractories, Glasses, and
Cobalt	Carbides
Copper	Cement
Copper "Benchmarks"	Trace Element Standards
Lead	Nuclear Materials
Magnesium	Special nuclear materials
Nickel	Plutonium assay
Nickel Superalloy, Trace Elements	Plutonium isotopic
Nickel oxide	Uranium assay
Selenium	Uranium isotopic
Tin	Neutron density standards
Titanium	Fission track glass standards
Zinc	Isotopic Reference Standards
Zirconium	
Nonferrous Alloys (solid form)	
Aluminum "Benchmarks"	
Copper	
Copper "Benchmarks"	
Lead	
Nickel	
Titanium	
Zinc	
Zirconium	

Table 1. continued.

CERTIFIED PHYSICAL PROPERTY STANDARDS

Ion Activity Standards

pH standards  
pD standards  
Ion selective electrodes

Mechanical and Metrology Standards

Magnification  
Coating thickness  
Glass  
Elasticity  
Density  
Polymer  
Rheology

Heat Standards

Superconductive thermometric fixed  
point devices  
Freezing Points

Defining fixed points  
Determined reference points

Melting points  
Calorimetric

Combustion  
Solution  
Heat source  
Enthalpy and heat capacity

Vapor pressure  
Thermal expansion  
Thermocouple materials  
Thermal resistance

Magnetic Standards

Magnetic susceptibility  
Magnetic moment  
Paramagnetic resonance

Optical Standards

Spectrophotometric  
Thermal emittance  
Refractive index

Radioactivity Standards

Alpha-particle standards  
Beta-particle and gamma-ray gas  
standards  
Alpha-particle, beta-particle,  
gamma-ray, and electron-capture  
solution standards  
Contemporary standard for carbon-14  
dating laboratories  
Environmental standards  
Low energy photon sources  
Gamma-ray "point-source" standards  
Radium gamma-ray solution standards  
Radium solution standards for  
random analysis  
Radioactivity standard reference  
materials currently not in stock

Metallurgical

Mössbauer

X-ray Diffraction

Gas Transmission

Permittivity

Reference Fuels

Resistivity

ENGINEERING TYPE STANDARDS

Standard Rubber and Rubber-  
Compounding Materials

Reference Magnetic Tapes

Centerline Drawings, OCR-B

Sizing Standards

Glass spheres for particle size  
Turbidimetric and fineness (cement)

RESEARCH MATERIALS

X-ray and Photographic Standards

Surface Flammability Standards

Smoke Density Chamber Standards

Water Vapor Permeance

Tape Adhesion Testing Standards

Color Standards

SPECIAL REFERENCE MATERIALS

The accurate determination of major and minor constituents in coals, ores, minerals, rocks, and refractories is important to our nation's scientific and industrial community as well as to commerce and trade. In many industries, traceability of quality control to the national measurement system is achieved through the use of Standard Reference Materials (SRM's). This publication is an attempt to describe, in general terms, the composition, certification, and use of such SRM's.

Essential information concerning the material composition, the certification parameters, and use is given in Table 2. Under "Remarks," additional data are provided.<sup>1</sup> All the data and information contained in this table were extracted from the Certificates or Certificates of Analysis issued for the included SRM's. An examination of the table gives the reader a general view of these SRM's. For more detailed information, the individual Certificates reproduced in Appendix II should be consulted as well as any references cited in each certificate. The composition of the same SRM's, excepting the coals, by chemical elements are listed in Table 3. In the tables, similar types of SRM's are grouped together to facilitate comparisons of their properties. The Certificates in Appendix II, however, are arranged in numerical order. The SRM's listed in the tables include all of the coal, ore, mineral, rock, and refractory standards that were in stock as of January 1, 1984. These SRM's are the result of the concerted efforts of a number of scientists from the NBS National Measurement Laboratory, Center for Analytical Chemistry, and from industry. Each Certificate lists the individuals and laboratories who contributed to the preparation and certification of the SRM.

In addition to the SRM's and their Certificates, NBS issues a series of Special Publications (SP), call the "260 Series," that relate directly to Standard Reference Materials as stated in the preface. The list of available publications in the "260 Series" is given at the beginning of this publication.<sup>2</sup>

Table 4 lists several NBS SP 260 publications directly related to the SRM's described in this work. They should be of value to the user of these particular materials. Also, NBS SP 260-54 contains in its appendices three reprints on: "Standard Quartz Cuvettes for High-Accuracy Spectrophotometry" (Appendix III); "Testing of Glass Volumetric Apparatus, 1959" (Appendix V); and "The Calibration of Small Volumetric Laboratory Glassware, 1974" (Appendix VI). The last two publications have been out of print for several years.

Other NBS publications, not in the "260 Series," and a number of NBS staff authored papers have been published that deal with specific SRM's or measurement techniques used in analytical chemistry. Some of these are: SP 148, The Role of Standard Reference Materials in Measurement Systems; SP 378, Accuracy in Spectrophotometry and Luminescence Measurements (255 pp., 1973); and SP 466, Standardization in Spectrophotometry and Luminescence Measurements (150 pp., 1977). NBS Monograph 100, Trace Characterization, Chemical and Physical (580 pp., 1967); SP 422, Accuracy in Trace Analysis, Sampling, Sample Handling, Analysis, Vol. 1 and 2 (1304 pp., 1976); and SP 464, Proceedings of the Eighth Materials Research Symposium, Methods and Standards for Environmental Measurements (691 pp., 1977), provide further information on methodology for the analysis of a variety of materials and the determination of numerous constituents. They all should be of particular interest to the analytical chemist.

<sup>1</sup>NOTE: The use of proprietary designations in Table 2 is for information only, and should not be construed as an endorsement of the product by either the Department of Commerce or the National Bureau of Standards. Table 3 was taken from an article by S. D. Raspberry published in Amer. Lab., 15 (5), 96 (1983).

<sup>2</sup>For complete bibliographic reference and ordering information, see "Other NBS Publications in This Series," pp. iv.



Table 2. Summary of Coal, Ore, Mineral, Rock, and Refractory Standards

## A. Coals

SRM	Material	Composition
1632b Trace Elements in Coal (Bituminous)	Obtained from Humphrey No. 7 mine and coal preparation plant of the Consolidation Coal Co., Christopher Coal Co. Div., Osage, W.V. The coal was reduced in size to -60 mesh and sieved prior to blending. The coal was then blended in a stainless steel coal blender.	Material should be vacuum dried at ambient temperature for 24 hours prior to use. Values based on a minimum sample size of 250 mg. C (total) 78.11; H 5.07; N 1.56; S 1.89; Volatile matter 35.4; Al 0.855; Ca .204; Fe .759; Mg .0383; K .0748; Na .0515; Ti .0454; values % by wt. As 3.72; Ba 67.5; Cd 0.0573; Co 2.29; Cu 6.28; Pb 3.67; Mn 12.4; Ni 6.10; Rb 5.05; Se 1.29; Th 1.342; U 0.436; Zn 11.89; values µg/g. Not certified: 17 additional constituents (see certificate).
1633a Trace Elements in Coal Fly Ash	Supplied by a coal fired power plant and is a product of Pennsylvania and West Virginia coals. Material was sieved through a #170 sieve and blended in a Vee blender.	Material should be dried to a constant weight before use. Values based on a 250-mg or more sample size. Ca 1.11; Fe 9.40; K 1.88; Mg 0.455; Na 0.17; Si 22.8, values % by wt. As 145; Cd 1.0; Cr 196; Cu 118; Hg 0.16; Ni 127; Pb 72.4; Rb 131; Se 10.3; Sr 830; Th 24.7; Tl 5.7; U 10.2; Zn 220, values µg/g. Not certified: 15 additional constituents (see certificate)
1635 Trace Elements in Coal (Sub-bituminous)	Provided by the Eagle Mine of the Imperial Coal Company, Erie, Colorado. Ground and sieved through a 230 µ sieve by the Colorado School of Mines Research Institute.	Material should be dried without heat to constant weight before use. Values based on 250-mg or more sample size. As 0.42; Cd 0.03; Cr 2.5; Cu 3.6; Pb 1.9; Mg 21.4; Ni 1.74; Se 0.9; Th 0.62; U 0.24; V 5.2; Zn 4.7, values µg/g. Fe 0.239; S 0.33, values % by wt. Not certified 10 additional constituents (see certificate).
2682 Sulfur in Coal (Sub-bituminous)	Obtained from Bell Ayr Mine, Gillette, Wyoming. The coal was reduced in size to -60 mesh and screened prior to blending. The -60 mesh coal was then blended in a stainless steel cone blender.	Sample vacuum dried at ambient temperature for 24 hours or oven drying for 24 hours at 105 °C. Values based on 250-mg or more sample size. Sulfur (wt. %) 0.47; Furnace Ash (wt. %) 6.37; Calorific Content 27.45 MJ.Kg <sup>-1</sup> . Not certified: 32 inorganic constituents (see certificate).

\*National Bureau of Standards, Center for Analytical Chemistry.

°American Society for Testing and Materials.



Certification	Use	Remarks
Analyses performed in the NBS CAC.* Estimated uncertainty 0.0008 TO 2.1 depending on constituent.	For the calibration of apparatus and the evaluation of techniques employed in the analysis of coal or similar materials.	Should be kept in its original bottle. Should not be exposed to intense sources of radiation, including ultraviolet lamps or sunlight.
Analyses performed in the NBS CAC. Estimated uncertainty 0.010 to 30 depending on constituent.	For the calibration of apparatus and methods used in analyses of coal fly ash and other materials with similar matrices for trace elements.	Should be kept in a tightly sealed bottle.
Analyses performed in the NBS CAC. Estimated uncertainty 0.15 to 1.5 depending on constituent.	Same as SRM 1632a.	Same as SRM 1632a.
Analyses performed in the Inorganic Analytical Research Division and Chemical Thermodynamics Division of NBS. Calorific values were determined by procedures recommended in standard ASTM <sup>®</sup> methods.	For the determination of sulfur in coal.	The Sulfur in Coal SRM's are sold individually rather than in sets; however, only one Certificate of Analysis is provided. Therefore, the user must be careful to use the data specific to the SRM being used. Material should be stored in a tightly sealed bottle away from sunlight and intense sources of radiation.

Table 2. A. Coals - continued.

SRM	Material	Composition
2683 Sulfur in Coal (Bituminous)	Obtained from Humphrey Mine, Osage, West Virginia. Processing same as SRM 2682.	Same as SRM 2682 except: Sulfur (wt. %) 1.85; Furnace Ash (wt. %) 6.85; Calorific Content 32.70 MJ.Kg <sup>-1</sup> .
2684 Sulfur in Coal (Bituminous)	Obtained from Delta Mine, Marion, Illinois. Processing same as SRM 2682.	Same as SRM 2682 except: Sulfur (wt. %) 3.00; Furnace Ash (wt. %) 11.09; Calorific Content 29.68 MJ.Kg <sup>-1</sup> .
2685 Sulfur in Coal (Bituminous)	Obtained from McElroy Mine, Captina, West Virginia. Processing same as SRM 2682.	Same as SRM 2682 except: Sulfur (wt. %) 4.62; Furnace Ash (wt. %) 16.53; Calorific Content 28.15 MJ.Kg <sup>-1</sup> .

## B. Rocks, Minerals, and Refractories

1c Argillaceous Limestone	Provided by Lone Star Ind., Inc., Cement and Construction Materials Group, Houston, Texas.	Limestone, ground, sieved, blended. Analyses performed on 0.5-g or more sample. Dried 2 hours at 110 °C. SiO <sub>2</sub> 6.84; Fe <sub>2</sub> O <sub>3</sub> 0.55; Al <sub>2</sub> O <sub>3</sub> 1.30; TiO <sub>2</sub> 0.07; P <sub>2</sub> O <sub>5</sub> 0.04; MnO 0.025; CaO 50.3; SrO 0.030; MgO 0.42; Na <sub>2</sub> O 0.02; K <sub>2</sub> O 0.28; loss on ignition 39.9; all values % by wt.
70a Feldspar	Feldspar, sample dried 2 hours at 105 °C.	SiO <sub>2</sub> 67.1; Al <sub>2</sub> O <sub>3</sub> 17.9; Fe <sub>2</sub> O <sub>3</sub> 0.07; TiO <sub>2</sub> 0.01; CaO 0.11; BaO 0.02; Na <sub>2</sub> O 2.5; K <sub>2</sub> O 11.8; Rb <sub>2</sub> O 0.06; loss on ignition 0.40; all values % by wt.
76a Burnt Refractory	Provided by Harbison-Walker Refractories Co., Garber Research Center, Pittsburgh, Pennsylvania. Dobies were air dried at 120 °C, then fired at 1427 °C for 10 hours. The ground material was converted to a fine powder (95%, 325 mesh) and was thoroughly mixed.	SiO <sub>2</sub> 54.9; Al <sub>2</sub> O <sub>3</sub> 38.7; Fe <sub>2</sub> O <sub>3</sub> 1.6; TiO <sub>2</sub> 2.0; ZrO <sub>2</sub> 0.15; MgO 0.52; CaO 0.22; K <sub>2</sub> O 1.33; Na <sub>2</sub> O 0.07; P <sub>2</sub> O <sub>5</sub> 0.12; Li <sub>2</sub> O 0.042; SrO 0.037; loss on ignition 0.34; all values % by wt.
77a Burnt Refractory	Same as SRM 76a.	SiO <sub>2</sub> 35.0; Al <sub>2</sub> O <sub>3</sub> 60.2; Fe <sub>2</sub> O <sub>3</sub> 1.0; TiO <sub>2</sub> 2.6; ZrO <sub>2</sub> 0.21; MgO 0.38; CaO 0.05; K <sub>2</sub> O 0.09; Na <sub>2</sub> O 0.037; P <sub>2</sub> O <sub>5</sub> 0.092; Li <sub>2</sub> O 0.02; SrO 0.009; loss on ignition 0.22; all values % by wt.

Certification	Use	Remarks
Same as SRM 2682.	Same as SRM 2682.	Same as SRM 2682.
Same as SRM 2682.	Same as SRM 2682.	Same as SRM 2682.
Same as SRM 2682.	Same as SRM 2682.	Same as SRM 2682.
Chemical analysis performed by eight cooperating laboratories including NBS-CAC. The estimated uncertainty: 0.005 to 0.3 depending on constituent.	For calibration and standardization of analytical methods and instrumentation.	Issued in cooperation with ASTM.
		Revision of Certificate dated 3-26-65.
Chemical analyses performed by three cooperating laboratories including NBS-CAC. The values are not expected to deviate from the accurate value by more than $\pm 1$ in the last significant figure reported.	For verifying the chemical and instrumental methods of analysis.	The loss on ignition is not a certified value but is given for information only. Issued in cooperation with ASTM.
Same as SRM 76a.	Same as SRM 76a.	Same as SRM 76a.

Table 2. B. Rocks, Minerals, and Refractories - continued.

SRM	Material	Composition
78a Burnt Refractory	Same as SRM 76a.	SiO <sub>2</sub> 19.4; Al <sub>2</sub> O <sub>3</sub> 71.7; Fe <sub>2</sub> O <sub>3</sub> 1.2; TiO <sub>2</sub> 3.2; ZrO <sub>2</sub> 0.31; MgO 0.70; CaO 0.11; K <sub>2</sub> O 1.22; Na <sub>2</sub> O 0.078; P <sub>2</sub> O <sub>5</sub> 1.3; Li <sub>2</sub> O 0.12; SrO 0.25; loss on ignition 0.42; all values % by wt.
81a Glass Sand	Ground glass sand powder 95% less than 106 $\mu$ m. Blended and dried at 105 °C for 2 hours before use.	Al <sub>2</sub> O <sub>3</sub> 0.66; Fe <sub>2</sub> O <sub>3</sub> 0.082; TiO <sub>2</sub> 0.12; ZrO <sub>2</sub> 0.034; all values % by wt. Cr <sub>2</sub> O <sub>3</sub> 46 $\mu$ g/g.
88a Dolomitic Limestone	Limestone	Sample dried 2 hours at 110 °C. SiO <sub>2</sub> 1.20; Al <sub>2</sub> O <sub>3</sub> 0.19; Fe <sub>2</sub> O <sub>3</sub> 0.28; TiO <sub>2</sub> 0.02; MnO 0.03; CaO 30.1; SrO 0.01; MgO 21.3; Na <sub>2</sub> O 0.01; K <sub>2</sub> O 0.12; P <sub>2</sub> O <sub>5</sub> 0.01; CO <sub>2</sub> 46.6; loss on ignition 46.7; all values expressed as percent.
97a Flint Clay	Provided by A. P. Green Fire Brick Co., Mexico, Missouri.	Sample dried 2 hours at 140 °C. SiO <sub>2</sub> 43.67; Al <sub>2</sub> O <sub>3</sub> 38.79; Fe <sub>2</sub> O <sub>3</sub> 0.45; TiO <sub>2</sub> 1.90; P <sub>2</sub> O <sub>5</sub> 0.36; K <sub>2</sub> O 0.50; Na <sub>2</sub> O 0.037; Li <sub>2</sub> O 0.11; BaO 0.07; MgO 0.15; CaO 0.11; SrO 0.18; Cr <sub>2</sub> O <sub>3</sub> 0.03; loss on ignition 13.32; all values are % by wt.
98a Plastic Clay	Same as SRM 97a.	Sample dried for 2 hours at 140 °C. SiO <sub>2</sub> 48.94; Al <sub>2</sub> O <sub>3</sub> 33.19; Fe <sub>2</sub> O <sub>3</sub> 1.34; TiO <sub>2</sub> 1.61; P <sub>2</sub> O <sub>5</sub> 0.11; K <sub>2</sub> O 1.04; Na <sub>2</sub> O 0.082; Li <sub>2</sub> O 0.070; BaO 0.03; MgO 0.42; CaO 0.31; SrO 0.039; Cr <sub>2</sub> O <sub>3</sub> 0.03; loss on igni- tion 12.44; all values % by wt.
99a Feldspar		Sample dried at 105 °C for 2 hours. SiO <sub>2</sub> 65.2; Al <sub>2</sub> O <sub>3</sub> 20.5; Fe <sub>2</sub> O <sub>3</sub> 0.06; TiO <sub>2</sub> 0.007; CaO 2.14; BaO 0.26; MgO 0.02; Na <sub>2</sub> O 6.2; K <sub>2</sub> O 5.2; P <sub>2</sub> O <sub>5</sub> 0.02; loss on ignition 0.26; all values are % by wt.

Certification	Use	Remarks
Same as SRM 76a.	Same as SRM 76a.	Same as SRM 76a.
Chemical analyses performed by eight cooperating laboratories including NBS-CAC.	Same as SRM 76a.	The recommended data is the best estimate of the true value based on data from the cooperators and NBS. Issued in cooperation with ASTM.
		Revision of Certificate dated 1-31-67.
Chemical analyses performed by three cooperating laboratories including NBS-CAC.	Same as SRM 76a.	ZrO <sub>2</sub> was determined by only one of the cooperating laboratories and found to be 0.063 % by wt.
Chemical analyses performed by three laboratories including NBS-CAC.	Same as SRM 76a.	ZrO <sub>2</sub> was determined by only one of the cooperating laboratories and found to be 0.042 % by wt.
		Revision of Certificate dated 3-26-65.



Table 2. B. Rocks, Minerals, and Refractories - continued.

SRM	Material	Composition
103a Chrome Refractory		Sample dried at 110 °C for 2 hours. Cr <sub>2</sub> O <sub>3</sub> 32.06; Al <sub>2</sub> O <sub>3</sub> 29.96; FeO 12.43; MnO 0.11; MgO 18.54; CaO 0.69; SiO <sub>2</sub> 4.63; TiO <sub>2</sub> 0.22; ZrO <sub>2</sub> 0.01; P <sub>2</sub> O <sub>5</sub> 0.01; all values are % by wt.
154b Titanium Dioxide	Prepared by the National Lead Industries, Research and Development Dept., St. Louis, Missouri.	TiO <sub>2</sub> 99.74% by wt. on samples dried 2 hours at 110 °C.
165a Glass Sand	Glass sand blended to insure homogeneity. Should be dried for 2 hours at 105 °C.	Al <sub>2</sub> O <sub>3</sub> 0.059; Fe <sub>2</sub> O <sub>3</sub> 0.012; TiO <sub>2</sub> 0.011; ZrO <sub>2</sub> 0.006; all values are % by wt.
198 Silica Brick		Al <sub>2</sub> O <sub>3</sub> 0.16; Fe <sub>2</sub> O <sub>3</sub> 0.66; TiO <sub>2</sub> 0.02; P <sub>2</sub> O <sub>5</sub> 0.022; MnO 0.008; CaO 2.71; MgO 0.07; Na <sub>2</sub> O 0.012; K <sub>2</sub> O 0.017; Li <sub>2</sub> O 0.001; loss on ignition 0.21; all values % by wt.
199 Silica Brick		Al <sub>2</sub> O <sub>3</sub> 0.48; Fe <sub>2</sub> O <sub>3</sub> 0.74; TiO <sub>2</sub> 0.06; P <sub>2</sub> O <sub>5</sub> 0.015; MnO 0.007; CaO 2.41; Na <sub>2</sub> O 0.015; K <sub>2</sub> O 0.094; Li <sub>2</sub> O 0.002; loss on ignition 0.17; all values % by wt.
278 Obsidian Rock	Natural glass obtained from Clear lake, Newberry Crater, Oregon, and processed by the Colorado School of Mines, Golden, Colorado, to a powder of <200 mesh.	The material should be dried to constant weight at 350-600 °C. Al <sub>2</sub> O <sub>3</sub> 14.15; CaO 0.983; FeO 1.36; Fe <sub>2</sub> O <sub>3</sub> 2.04; K <sub>2</sub> O 4.16; MnO 0.052; Na <sub>2</sub> O 4.84; P <sub>2</sub> O <sub>5</sub> 0.036; SiO <sub>2</sub> 73.05; TiO <sub>2</sub> 0.245; all data wt %. Cu 3.9; Ni 3.6; Rb 127.5; Sr 63.5; Th 12.4; Tl 0.54; U 4.58; Pb 16.4; data wt (µg/g).

Certification	Use	Remarks
Chemical analyses performed by eight cooperating laboratories including NBS-CAC.	Certified primarily for use in the analysis of ores and geological materials containing chromium.	For additional information on methods used for analysis and certification of constituent elements see certificate.
Analyses performed by three cooperating laboratories including NBS-CAC. $\text{TiO}_2$ was determined by controlled-potential coulometry and by volumetry. The uncertainty is 0.05 and represented the 95% confidence interval of the mean of all accepted values.	Certified primarily for application in the paint and ceramic industries.	For additional information on the material and its composition see Certificate.
Chemical analyses performed by eight cooperating laboratories including NBS-CAC.		The certified data is the best estimate of the true value based on the measurements from the cooperators and NBS. SRM 165a was issued in cooperation with ASTM.
Chemical analyses performed by twelve cooperating laboratories including NBS-CAC. All results are based on samples dried at 105-110 °C.		One cooperating laboratory has reported a content of less than 0.01 $\text{ZrO}_2$ % by wt.
Same as SRM 198.		One cooperating laboratory has reported a content of 0.01 0.01 $\text{ZrO}_2$ % by wt.
Chemical analyses performed by four cooperating laboratories including NBS-CAC. Uncertainty of measurements varies from $\pm 0.3$ to 0.002 depending on element.	For evaluation of the accuracy of analytical methods and instrumentation used in the analysis of geological type materials.	Additional 21 constituents given but not certified (see certificate).

Table 2. B. Rocks, Minerals, and Refractories - continued.

SRM	Material	Composition
688 Basalt Rock	Obtained from a Cenozoic basalt flow near Jackpot, Nevada, and processed by the Colorado School of Mines, Golden, Colorado.	Sample dried at 105 °C for 24 hours. $Al_2O_3$ 17.36; FeO 7.64; $Fe_2O_3$ 10.35; $K_2O$ 0.187; MnO 0.167; $Na_2O$ 2.15; $P_2O_5$ 0.134; $SiO_2$ 48.47; $TiO_2$ 1.17, all % by wt. Cr 332; Rb 1.91; Sr 169.2 Th 0.33; Pb 3.3, all $\mu g/g$ .
C. Ores		
27f Iron Ore (Sibley)	The iron ore was provided by the U.S. Steel Corp., Pittsburgh, Pennsylvania. The ore was crushed, dry ground, and sieved at the Colorado School of Mine Research Institute, Golden, Colorado, to pass a 150 mesh sieve with about 50% passing a 200 mesh sieve. Analytical samples were dried 1 hour at 105 °C.	Total Fe 65.97; $SiO_2$ 4.17; $Al_2O_3$ 0.82; P 0.041; S 0.005; $TiO_2$ 0.019; MnO 0.011; CaO 0.039; MgO 0.019; $Na_2O$ 0.012; $K_2O$ 0.008; all values % by wt.
69b Bauxite (Arkansas)	Provided by the Aluminum Company of America, Bauxite, Arkansas. The material was processed at the Colorado School of Mine Research Institute. Mineralogical composition: 30% kaolinite, 60% gibbsite, 10% siderite (semiquantitative).	The bauxite powder (<0.08 mm) was dried at 140 °C for 2 hours. Analytical sample of 1-g or more. $Al_2O_3$ 48.8; $Fe_2O_3$ 7.14; $SiO_2$ 13.43; $TiO_2$ 1.90; $ZrO_2$ 0.29; $P_2O_5$ 0.118; $V_2O_5$ 0.028; $Cr_2O_3$ 0.011; CaO 0.13; MgO 0.085; MnO 0.110; ZnO 0.0035; $K_2O$ 0.068; $SO_2$ 0.63; loss of ignition 27.2 at 1050 °C; all values % by wt. Estimated uncertainty 0.0005 to 0.2 depending on element.
79a Fluorspar	Provided by American Smelting and Refining Co., El Paso, Texas, and ground to pass an 80 mesh screen.	$CaF_2$ 97.39 $\pm$ 0.06 % by wt.

Certification	Use	Remarks
Analyses performed by three cooperating laboratories including NBS-CAC. Uncertainties for % by wt. constituents: from 0.1 to 0.002; for $\mu\text{g/g}$ constituents: 9 to 0.01.	For verifying the accuracy of chemical and instrumental methods of analysis used in the analysis of geological type materials.	
Chemical analyses performed by seven cooperating laboratories including NBS-CAC using the methods described in Part 12, Chemical Analysis of Metals and Metal Bearing Ores, Annual Book of ASTM Standards.	For use in verifying chemical methods of analysis and in calibrating instrumental methods of analysis.	Issued in cooperation with ASTM.
Chemical analyses performed by eleven cooperating laboratories including NBS-CAC.	For verification of chemicals and instrumental methods of analysis.	Additional composition (not certified): BaO (0.008); $\text{Na}_2\text{O}$ (0.025); Ce (0.024); Co (0.0001); Hf (0.0063); Sc (0.0008). Issued in cooperation with ASTM.
Chemical analyses performed in cooperation with the U.S. Customs Laboratories, Division of Technical Services, following a standard procedure described in detail in the Certificate.	To assay imported fluorspar for industrial applications.	Additional percent composition (not certified): Fe (0.05-0.1) Al and Sr (0.01-0.1); Mg (0.01-0.05); Na (0.001-0.01); Ba (0.001-0.005); K (<0.005); Cu, Li, Mn, Pb, and Ti (<0.001); $\text{SiO}_2$ (0.67); all are % values. <sup>2</sup> Revision of Certificate dated 12-6-71.

Table 2. C. Ores - continued.

SRM	Material	Composition
113a Zinc Concentrate	Provided by Cominco American Inc., Spokane, Washington, and prepared at the Magmont Mines, Bixby, Missouri, in the form of powder (<0.15 mm).	Sample dried 1 hour at 105 °C. Zn 57.3; Co (0.11); Ni (0.07); S 30.6; Cd 0.78; Cu 0.31; Pb 2.80; Fe 2.08; CaO 1.1; MgO 0.75; SiO <sub>2</sub> (1.54); Ag 0.046, all values are % by wt.
120b Phosphate Rock (Florida)	Provided by American Cyanamid Co. as a powder to pass 200 mesh sieve.	Sample dried 1 hour at 105 °C. P <sub>2</sub> O <sub>5</sub> 34.57; CaO 49.40; SiO <sub>2</sub> 4.68; F 3.84; Fe <sub>2</sub> O <sub>3</sub> 1.10; Al <sub>2</sub> O <sub>3</sub> 1.06; MgO 0.28; Na <sub>2</sub> O 0.35; MnO 0.032; K <sub>2</sub> O 0.12-0.090; TiO <sub>2</sub> 0.15; CO <sub>2</sub> 2.79; CdO 0.002; all values are % by wt. Uranium 128.4 µg/g.
180 High-Grade Fluorspar	Provided by Minera Frisco of San Francisco Del Oro, Chih., Mexico.	CaF <sub>2</sub> 98.80 ± 0.03 % by wt.
181, 182, 183 Lithium Ores	Fine powder of spodumene (SRM 181), petalite (SRM 182), and lepidolite (SRM 183).	Li <sub>2</sub> O wt. % in SRM 181 - 6.3 <sub>9</sub> ; SRM 182 - 4.3 <sub>4</sub> ; and SRM 183 - 4.1 <sub>2</sub> .
277 Tungsten Concentrate	Provided by GTE Sylvania, Towanda, Pennsylvania, as a powder sieved (<0.15 mm) and blended. It is a mixture of concentrates from China, Thailand, and USA.	WO <sub>3</sub> - 67.4 wt. %, determined on 1-g or more sample.
329 Zinc Concentrate	Provided by Cominco American Inc., Spokane, Washington, and prepared at the Sullivan Mine, Trail, B.C., Canada, in the form of powder (<0.15 mm).	Sample dried 1 hour at 105 °C. Zn 45.5; Pb 6.06; Fe 12.94; CaO 0.08; MgO 0.16; Cd 0.14; Cu 0.13; Co (0.009); Ni (0.006); S (31.7); SiO <sub>2</sub> (0.61) In 0.019; Ag 0.0089; all values are % by wt.



Certification	Use	Remarks
Chemical analyses performed by four cooperating laboratories including NBS-CAC. The data are not expected to deviate from the true value by more than $\pm 1$ in the last significant figures reported.	Intended for the verification of chemical and instrumental methods of analysis.	Figures in parentheses are not certified. Should be kept tightly closed except when in direct use. Store in a desiccator over desiccant.
Chemical analyses performed by six cooperating laboratories including NBS-CAC. Uranium was determined at NBS by thermal ionization mass spectrometry.	For the verification of chemical optical emission and x-ray spectrometric methods of analysis.	For potassium values see Certificate.
Additional trace elements detected but not certified: Fe 0.1-1.0; Al, Ba, Mg, Pb, Si, and Sr 0.01-0.1; Cu, K, Mn, Na, Ti, and V 0.001-0.01; Ag and Li less than 0.001; all values expressed in percent.	Issued primarily for geochemical use.	Detailed information on the method used for the determination of $\text{CaF}_2$ is given in the Certificate.
The deviation from given values is not expected to be more than $\pm 5$ subscript number.	For verifying the accuracy of assay methods.	Revision of Certificate dated 2-24-58.
Chemical analyses performed in cooperation with 13 laboratories including NBS-CAC. The estimated uncertainty 0.3 wt. %. Additional elements indicated but not certified are: Ca, Fe, Pb, Mn, Nb, P, Si, S, Sn, Ti, As, Mo, Bi, Ce, Cr, Cu, Cd, La, Nd, O, Sc, Ta, Th, U, Y, and Zr.	To verify the accuracy of chemical and instrumental methods of analysis.	Issued in cooperation with ASTM.
Chemical analyses performed by four cooperating laboratories including NBS-CAC. The data are not expected to deviate from the true value by more than $\pm 1$ in the last significant figure reported.	Intended for the verification of chemical and instrumental methods of analysis.	Figures in parentheses are not certified.

Table 2. C. Ores - continued.

SRM	Material	Composition
330 Copper Ore, Mill Heads	Provided by Magma Copper Co., San Manuel, Arizona. It was sieved and blended at NBS.	Sample dried at 105 °C for 2 hours. Cu 0.84; Mo 0.018; both values % by wt. Re 0.30 ppm by wt. Not certified: Au (0.093); Ag (1.51) ppm by wt.
331 Copper Ore, Mill Tails	Provided by Magma Copper Co., San Manuel, Arizona.	Cu 0.091; Mo 0.0022; both % by wt. Re 0.04 ppm by wt. on samples dried at 105 °C for 2 hours. Not certified: Au (0.034); Ag (0.243) ppm by wt.
332 Copper Concentrate	Provided by Magma Copper Co., San Manuel, Arizona.	Cu 28.4; Mo 0.64 % by wt. Re 10.2 ppm by wt. on samples dried at 105 °C for 2 hours. Not certified: Au (2.14); Ag (38.7) ppm by wt.
333 Molybdenum Concentrate	Provided by Magma Copper Co., San Manuel, Arizona. Homogeneity obtained by special blending and mixing procedures.	Cu 1.038; Mo 55.3; Re 0.087; all % by wt. on samples "as received." Not certified: Au (8.9); Ag (25.0) ppm by wt.
690 Iron Ore Concentrate (Canada)	Provided by the Iron Ore Company of Canada, Labrador City, Newfoundland, Canada, as a powder (<0.1 mm).	Fe 66.85; SiO <sub>2</sub> 3.71; Al <sub>2</sub> O <sub>3</sub> 0.18; P 0.011; S 0.003; TiO <sub>2</sub> 0.022; MnO 0.23; CaO 0.20; MgO 0.18; Na <sub>2</sub> O 0.003; K <sub>2</sub> O 0.0030, on samples dried at 105 °C for 1 hours. All values are % by wt.
691 Reduced Iron Oxide	Provided by Allis-Chalmers, Reduction Systems Div., Milwaukee, Wisconsin, and processed as a powder (~200 mesh) at the Colorado School of Mines Research Institute, Golden, Colorado.	Values based on 0.5-g or more sample size. Fe, total 90.8; Fe, metallic 84.6; SiO <sub>2</sub> 3.7; Al <sub>2</sub> O <sub>3</sub> 1.22; TiO <sub>2</sub> 0.27; CaO 0.63; MnO 0.043; MgO 0.52; Na <sub>2</sub> O 0.186; Cu 0.032; Co 0.030; P 0.006; S 0.008; C 0.12; all values % by wt. Not certified 11 additional constituents (see certificate).
692 Iron Ore (Labrador)	Prepared by the Bethlehem Steel Corp., Bethlehem, Pennsylvania, as a fine powder (200 mesh).	Sample dried 1 hour at 105 °C, values based on 0.5-g or more sample. Fe 59.58; SiO <sub>2</sub> 10.14; Al <sub>2</sub> O <sub>3</sub> 1.41; P 0.039; S <sup>0</sup> 0.005; TiO <sub>2</sub> 0.045; MnO 0.46; CaO 0.023; MgO 0.035; Na <sub>2</sub> O 0.008; K <sub>2</sub> O 0.039, all values % by wt.

Certification	Use	Remarks
Analyses performed by two cooperating laboratories including NBS-CAC. Estimated uncertainty 0.01 and 0.001 for Cu and Mo, and 0.06 for Re.	For verifying the accuracy of chemical and instrumental methods of analysis, and for evaluation of the material balance in copper mining and metallurgical industries.	Keep the material tightly closed in a desiccator over desiccant. Revision of Certificate of 2-20-73.
Estimated uncertainty 0.001 for Cu; 0.0002 for Mo; and 0.02 for Re.	Same as SRM 330.	Same as SRM 330.
Estimated uncertainty 0.1 for Cu; 0.01 for Mo; and 0.2 for Re.	Same as SRM 330.	Same as SRM 330, except revision of Certificate dated 2-20-73 and 1-20-77.
Analyses performed by two cooperating laboratories including NBS-CAC. Estimated uncertainty 0.010 for Cu; 0.1 for Mo; 0.001 for Re.	Same as SRM 330.	Same as SRM 330.
Analyses performed by six cooperating laboratories including NBS-CAC. Estimated uncertainty from 0.0005 to 0.01 depending on constituent.	For verifying the chemical and instrumental methods of analysis.	Issued in cooperation with ASTM.
Analyses performed by six cooperating laboratories including NBS-CAC. Estimated uncertainty 0.001 to 0.6 depending on constituent.	For the evaluation of chemical and spectroscopic methods of analysis.	Material packaged in dry nitrogen to prevent oxidation. Should be stored in desiccator over desiccant. Issued in cooperation with ASTM.
Analyses performed by six cooperating laboratories including NBS-CAC. Estimated uncertainty 0.001 to 0.06 depending on constituent.	For verifying the chemical and instrumental methods of analysis.	Issued in cooperation with ASTM.

Table 2. C. Ores - continued.

SRM	Material	Composition
693 Iron Ore (Nimba)	Prepared by the Bethlehem Steel Corp., Bethlehem, Pennsylvania, as a fine powder (200 mesh).	Sample dried 1 hour at 105 °C, values based on 0.5-g or more sample. Fe 65.11; SiO <sub>2</sub> 3.87; Al <sub>2</sub> O <sub>3</sub> 1.02; P 0.056; S 0.005; TiO <sub>2</sub> 0.035; MnO 0.091; CaO 0.016; MgO 0.013; Na <sub>2</sub> O 0.0028; K <sub>2</sub> O 0.0028; all values % by wt
696 Bauxite (Surinam)	Same as SRM 69b. The material was mined in Surinam, South America. Mineralogical composition: 5% kaolinite, 80% gibbsite, 10% pyrite, 5% anatase (semiquantitative).	The bauxite powder (<0.08 mm) was dried at 140 °C for 2 hours. Analytical sample of 1-g or more. Al <sub>2</sub> O <sub>3</sub> 54.5; Fe <sub>2</sub> O <sub>3</sub> 8.70; SiO <sub>2</sub> 3.79; TiO <sub>2</sub> 2.64; ZrO <sub>2</sub> 0.14; P <sub>2</sub> O <sub>5</sub> 0.050; V <sub>2</sub> O <sub>5</sub> 0.072; Cr <sub>2</sub> O <sub>3</sub> 0.047; CaO 0.018; MgO 0.012; MnO 0.004; ZnO 0.0014; K <sub>2</sub> O 0.009; SO <sub>3</sub> 0.21; loss on ignition 29.9 at 1050 °C; all values % by wt. Estimated uncertainty 0.0007 to 0.3 depending on element.
697 Bauxite (Dominican)	Same as SRM 69b. The material was mined in the Dominican Republic. Mineralogical composition: 15% kaolinite, 50% gibbsite, 10% boehmite, 5% anatase; 20% hematite (semiquantitative).	The bauxite powder (<0.08 mm) was dried at 140 °C for 2 hours. Analytical sample of 1-g or more. Al <sub>2</sub> O <sub>3</sub> 45.8; Fe <sub>2</sub> O <sub>3</sub> 20.0; SiO <sub>2</sub> 6.81; TiO <sub>2</sub> 2.52; ZrO <sub>2</sub> 0.065; P <sub>2</sub> O <sub>5</sub> 0.97; V <sub>2</sub> O <sub>5</sub> 0.063; Cr <sub>2</sub> O <sub>3</sub> 0.100; CaO 0.771; MgO 0.18; MnO 0.41; ZnO 0.037; K <sub>2</sub> O 0.062; SO <sub>3</sub> 0.13; loss on ignition 22.1 at 1050 °C. All values % by wt. Estimated uncertainty 0.2 to 0.003 depending on element.
698 Bauxite (Jamaican)	Mined in Jamaica, provided by Reynolds Metals Co., Bauxite, Arkansas, processed at the Colorado School of Mines Research Institute. Mineralogical composition: 75% gibbsite, 20% hematite, 5% anatase (semiquantitative).	The bauxite powder (<0.08 mm) was dried at 140 °C for 2 hours. Analytical sample of 1-g or more. Al <sub>2</sub> O <sub>3</sub> 48.2; Fe <sub>2</sub> O <sub>3</sub> 19.6; SiO <sub>2</sub> 0.69; TiO <sub>2</sub> 2.38; ZrO <sub>2</sub> 0.061; P <sub>2</sub> O <sub>5</sub> 0.37; V <sub>2</sub> O <sub>5</sub> 0.064; Cr <sub>2</sub> O <sub>3</sub> 0.080; CaO 0.62; MgO 0.058; MnO 0.38; ZnO 0.029; K <sub>2</sub> O 0.010; SO <sub>3</sub> 0.22; loss on ignition 27.3 at 1050 °C. All values % by wt. Estimated uncertainty 0.002 to 0.4 depending on element.

Certification	Use	Remarks
Analyses performed in cooperation with five laboratories including NBS-CAC. Estimated uncertainty 0.0005 to 0.07 depending on constituent.	Same as SRM 692.	Same as SRM 692.
Same as SRM 69b.	Same as SRM 69b.	Additional composition (not certified): BaO (0.004); Na <sub>2</sub> O (0.007); Ce (0.0041); Co <sup>2+</sup> (0.00009); Hf (0.0032); Sc (0.0008). Issued in cooperation with ASTM.
Same as SRM 69b.	Same as SRM 69b.	Additional composition (not certified): BaO (0.015); Na <sub>2</sub> O (0.036); Ce (0.069); Co <sup>2+</sup> (0.0013); Hf (0.0014); Sc (0.0058). Issued in cooperation with ASTM.
Same as SRM 69b.	Same as SRM 69b.	Additional composition (not certified): BaO (0.008); Na <sub>2</sub> O (0.015); Ce (0.030); Co <sup>2+</sup> (0.0045); Hf (0.0015); Sc (0.0051). Issued in cooperation with ASTM.

Table 3. Composition of SRM's Prepared from Ores, Minerals, Rocks, and Refractories (listed by chemical constituents and category).

Constituents	Composition of ore SRMs <sup>a</sup>					
	271 Iron ore, Sibley, 100 g	690 Iron ore, Canada, 150 g	691 Iron oxide, reduced, 100 g	692 Iron ore, Labrador, 150 g	693 Iron ore, Nimba, 150 g	69b Bauxite, Ark., 60 g
Al <sub>2</sub> O <sub>3</sub>	0.82	0.18	1.22	1.41	1.02	48.8
BaO	—	—	—	—	—	(0.008) <sup>b</sup>
C (Total)	—	—	.12	—	—	—
Cd	—	—	—	—	—	—
CdO	—	—	—	—	—	—
CaO	.039	.20	.63	.023	.016	.13
Co	—	—	.030	—	—	(.0001)
CO <sub>2</sub>	—	—	—	—	—	—
Cu	—	—	.032	—	—	—
Cr <sub>2</sub> O <sub>3</sub>	—	—	—	—	—	.011
F	—	—	—	—	—	—
In	—	—	—	—	—	—
Hf	—	—	—	—	—	(.0063)
Fe (Total)	65.97	66.85	90.8	59.58	65.11	—
Fe <sub>2</sub> O <sub>3</sub>	—	—	—	—	—	7.14
Pb	—	—	—	—	—	—
Ce	—	—	—	—	—	(0.024)
MgO	0.019	0.18	0.52	0.035	0.013	.085
MnO	.011	.23	.043	.46	.091	.110
P	.041	.011	.006	.039	.056	—
Ni	—	—	—	—	—	—
P <sub>2</sub> O <sub>5</sub>	—	—	—	—	—	.118
K <sub>2</sub> O	.008	.0030	—	.039	.0028	.068
SiO <sub>2</sub>	4.17	3.71	3.7	10.14	3.87	13.43
Ag	—	—	—	—	—	—
Na <sub>2</sub> O	0.012	0.003	0.186	0.008	0.0028	(0.025)
S	.005	.003	.008	.005	.005	—
Sc	—	—	—	—	—	(.0008)
SO <sub>2</sub>	—	—	—	—	—	.63
TiO <sub>2</sub>	.019	.022	.27	.045	.035	1.90
U	—	—	—	—	—	—
V <sub>2</sub> O <sub>5</sub>	—	—	—	—	—	0.028
WO <sub>3</sub>	—	—	—	—	—	—
Zn	—	—	—	—	—	—
ZnO	—	—	—	—	—	.0035
ZrO <sub>2</sub>	—	—	—	—	—	.29
Loss on ignition	—	—	—	—	—	27.2
Moisture	—	—	—	—	—	—

Constituents	SRM type, wt/unit						
	696 Bauxite, Surinam, 60 g	697 Bauxite, Dominican, 60 g	698 Bauxite, Jamaican, 60 g	120b Phosphate rock, Fla., 90 g	277 Tungsten conc., 100 g	113a Zinc ore conc., 100 g	329 Zinc conc., 100 g
Al <sub>2</sub> O <sub>3</sub>	54.5	45.8	48.2	1.06	—	—	—
BaO	(0.004)	(0.015)	(0.008)	—	Ta (0.20)	—	—
C (Total)	—	—	—	—	—	—	—
Cd	—	—	—	—	—	0.78	0.14
CdO	—	—	—	0.002	—	—	—
CaO	0.18	.71	.62	49.40	Ca (.37)	1.1 <sub>9</sub>	.08
Co	(.00009)	(.0013)	(.0045)	—	—	(0.11)	(.009)
CO <sub>2</sub>	—	—	—	2.79	—	—	—
Cu	—	—	—	—	—	0.31	13 <sub>2</sub>
Cr <sub>2</sub> O <sub>3</sub>	.047	.100	.080	—	Nb (1.00)	—	—
F	—	—	—	3.84	—	—	—
In	—	—	—	—	—	—	.019
Hf	(.0032)	(.0014)	(.0015)	—	—	—	—
Fe (Total)	—	—	—	—	(7.4)	2.08	12.9 <sub>4</sub>
Fe <sub>2</sub> O <sub>3</sub>	8.70	20.0	19.6	1.10	—	—	—
Pb	—	—	—	—	(0.07)	2.80	6.0 <sub>6</sub>
Ce	(0.0041)	(0.069)	(0.030)	—	—	—	—
MgO	.012	.18	.058	0.28	—	0.75	0.16 <sub>5</sub>
MnO	.004	.41	.38	.032	Mn (10.0)	—	—
P	—	—	—	—	(0.03)	—	—
Ni	—	—	—	—	—	(.07)	(.006 )
P <sub>2</sub> O <sub>5</sub>	.050	.97	.37	34.57	—	—	—
K <sub>2</sub> O	.009	.062	.010	0.12	—	—	—
SiO <sub>2</sub>	3.79	6.81	.69	4.68	Si (.85)	(1.54)	(.61)
Ag	—	—	—	—	—	0.046 <sub>7</sub>	.0089
Na <sub>2</sub> O	(0.007)	(0.036)	(.015)	0.35	—	—	—
S	—	—	—	—	(.25)	30.6	(31.7)
Sc	(.0008)	(.0058)	(.0051)	—	—	—	—
SO <sub>2</sub>	.21	.13	.22	—	O <sub>2</sub> (21.4)	—	—
TiO <sub>2</sub>	2.64	2.52	2.38	.15	Ti (2.2)	—	—
U	—	—	—	128.4 µg/g	—	—	—
V <sub>2</sub> O <sub>5</sub>	0.072	0.063	0.064	—	Mo (0.06)	—	—
WO <sub>3</sub>	—	—	—	—	67.4	—	—
Zn	—	—	—	—	—	57.3	45.5
ZnO	.0014	.037	.029	—	Sn (0.54)	—	—
ZrO <sub>2</sub>	.14	.065	.061	—	—	—	—
Loss on ignition	29.9	22.1	27.3	—	—	—	—
Moisture	—	—	—	—	—	0.08	0.4 <sub>5</sub>

\*Concentrations expressed in wt% unless noted otherwise.

\*\*Values in parentheses are not certified, but are given for information only.



Composition of rock, mineral, and refractory SRMs<sup>a</sup>

Constituents	SRM type, wt/unit							
	1c Limestone, argillaceous, 50 g	88a Limestone, dolomitic, 50 g	70a Feldspar, potash, 40 g	99a Feldspar, soda, 40 g	97e Clay, flint, 60 g	98a Clay, plastic, 60 g	81a Glass sand, 75 g	165a Glass sand, (low iron), 75 g
Al <sub>2</sub> O <sub>3</sub>	1.30	0.19	17.9	20.5	38.79	33.19	0.66	0.059
BaO	—	—	0.02	0.26	0.07 <sub>5</sub>	0.03	—	—
CaO	50.3	30.1 <sub>5</sub>	.11	2.14	.11	.31	—	—
Cr <sub>2</sub> O <sub>3</sub>	—	—	—	—	.03	.03	46 µg/g	(1.1) <sup>b</sup> µg/g
CO <sub>2</sub>	—	46.6	—	—	—	—	—	—
FeO	—	—	—	—	—	—	—	—
Fe <sub>2</sub> O <sub>3</sub>	0.55	0.28	0.07 <sub>5</sub>	0.06 <sub>5</sub>	0.45	1.34	0.082	0.012
Li <sub>2</sub> O	—	—	—	—	.11	0.070	—	—
MgO	0.42	21.3	—	.02	.15	.42	—	—
MnO	.025	0.03	—	—	—	—	—	—
P <sub>2</sub> O <sub>5</sub>	.04	.01	—	.02	.36	.11	—	—
K <sub>2</sub> O	.28	.12	11.8	5.2	.50	1.04	—	—
Rb <sub>2</sub> O	—	—	0.06	—	—	—	—	—
SiO <sub>2</sub>	6.84	1.20	67.1	65.2	43.67	48.94	—	—
Na <sub>2</sub> O	0.02	0.01	2.5 <sub>6</sub>	6.2	0.037	0.082	—	—
SrO	.030	.010	—	—	.18	.039	—	—
TiO <sub>2</sub>	.07	.02	0.10	0.007	1.90	1.61	.12	.011
ZrO <sub>2</sub>	—	—	—	—	—	—	.034	.006
Loss on ignition	39.9	46.7	.40	.26	13.32	12.44	—	—

	154b	278	688	SRM type, wt/unit					
	Titanium dioxide, 90 g	Obsidian rock, 35 g	Basalt rock, 60 g	76a Bunt refractory, (Al <sub>2</sub> O <sub>3</sub> —40%) 75 g	77a Bunt refractory, (Al <sub>2</sub> O <sub>3</sub> —60%) 75 g	78a Bunt refractory, (Al <sub>2</sub> O <sub>3</sub> —70%) 75 g	103a Chrome refractory, 60 g	198 Silica refractory, 45 g	199 Silica refractory, 45 g
Al <sub>2</sub> O <sub>3</sub>	—	14.15	17.36	38.7	60.2	71.7	29.96	0.16	0.48
BaO	—	—	—	—	—	—	—	—	—
CaO	—	0.983	—	0.22	0.05	0.11	0.69	2.71	2.41
Cr <sub>2</sub> O <sub>3</sub>	—	—	—	—	—	—	32.06	—	—
CO <sub>2</sub>	—	—	—	—	—	—	—	—	—
FeO	—	—	—	—	—	—	12.43	—	—
Fe <sub>2</sub> O <sub>3</sub>	—	2.04	10.35	1.6 <sub>0</sub>	1.0 <sub>0</sub>	1.2	—	0.66	0.74
Li <sub>2</sub> O	—	—	—	0.042	0.2 <sub>5</sub>	0.12	—	.001	.002
MgO	—	—	—	.52	.38	.70	18.54	.07	.13
MnO	—	0.052	0.167	—	—	—	0.11	.008	.007
P <sub>2</sub> O <sub>5</sub>	—	.036	.134	0.12 <sub>0</sub>	0.092	1.3	.01	.022	.015
K <sub>2</sub> O	—	4.16	.187	1.33	.09 <sub>0</sub>	1.22	—	.017	.094
Rb <sub>2</sub> O	—	—	—	—	—	—	—	—	—
SiO <sub>2</sub>	—	73.05	48.4	54.9	35.0	19.4	4.63	—	—
Na <sub>2</sub> O	—	4.84	2.15	0.07	0.037	0.078	—	.012	.015
SrO	—	—	—	.037	.009	.25	—	—	—
TiO <sub>2</sub>	99.74	0.245	1.17	2.0 <sub>3</sub>	2.6 <sub>8</sub>	3.2 <sub>2</sub>	0.22	.02	.06
ZrO <sub>2</sub>	—	—	—	0.15	0.21	0.31	0.01	—	—
Loss on ignition	—	—	—	(0.34)	(0.22)	(0.42)	—	.21	.17

<sup>a</sup>Concentrations expressed in wt% unless noted otherwise.<sup>b</sup>Numbers in parentheses are not certified, but are given for information only.

Composition of ore standard reference materials <sup>a</sup>

Constituents	SRM, Type, wt/unit								
	79a Fluorspar, customs grade, 120 g	180 Fluorspar, high grade, 120 g	181 Lithium ore (spodumene), 45 g	182 Lithium ore (petalite), 45 g	183 Lithium ore (lepidolite), 45 g	330 Copper ore mill heads, 100 g	331 Copper ore mill tails, 100 g	332 Copper conc., 50 g	333 Molybdenum conc., 35 g
CaF <sub>2</sub>	97.39	98.80	—	—	—	—	—	—	—
Li <sub>2</sub> O	—	—	6.3 <sub>g</sub>	4.3	4.1	—	—	—	—
Cu	—	—	—	—	—	0.84	0.091	28.4	1.038
Re	—	—	—	—	—	30 ppm	04 ppm	10.2 ppm	987
Mo	—	—	—	—	—	.018	.0022	.64	55.3
Au	—	—	—	—	—	(0.93) ppm <sup>b</sup>	(.034) ppm	(2.14) ppm	(8.9) ppm
Ag	—	—	—	—	—	(1.51) ppm	(.243) ppm	(38.7) ppm	(25) ppm

<sup>a</sup>Concentrations expressed in wt percent unless noted otherwise.<sup>b</sup>Values in parentheses are not certified, but are given for information only.

Table 4. NBS Publications in the "260 Series" Related to Coal, Ore,  
Mineral, Rock, and Refractory Standards

<u>Publication</u>	<u>Title</u>	<u>SRM No.</u>
260-8	Analysis of Uranium Concentrates at the National Bureau of Standards	
260-37	Methods of Analysis of NBS Clay Standards	97a, 98a
260-94	Methods and Procedures Used at the National Bureau of Standards to Certify Sulfur in Coal SRM's for Sulfur Content, Calorific Value, Ash Content	2682, 2683, 2684, 2685

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# Appendix I.

## Alphabetical Index by Standard Reference Material Name

Name	SRM
Acetanilide	141c
Acid Open-Hearth Steel, 0.2% Carbon	19G
Acid Potassium Phthalate	84j
AISI 1045 Steel	20g
AISI 4340 Steel	36l
AISI 4340 Steel	1261a
AISI 94B17 Steel (Modified)	362
AISI 94B17 Steel (Modified)	1262a
Albacore Tuna	RM 50
Alkali Lead Silicate Glass	712
Alpha Quartz	1878
Alumina (Reduction Grade)	699
Alumina Silicate Glass	714
Aluminosilicate Glass	715
Aluminum Alloy	85B
Aluminum Alloy 6011 (Modified)	858
Aluminum Alloy 6011 (Modified)	1258
Aluminum Alloy 7075	859
Aluminum Alloy 7075	1259
Aluminum Block, Eddy Current Conductivity	1860
Aluminum Block, Eddy Current Conductivity	1861
Aluminum Block, Eddy Current Conductivity	1862
Aluminum Block, Eddy Current Conductivity	1863
Aluminum Brass Standard for Optical Emission and X-ray Spectroscopic Analysis	1118
Aluminum Brass Standard for Optical Emission and X-ray Spectroscopic Analysis	C1118
Aluminum Brass Standard for Optical Emission and X-ray Spectroscopic Analysis	1119
Aluminum Brass Standard for Optical Emission and X-ray Spectroscopic Analysis	C1119
Aluminum Casting Alloy 356	855
Aluminum Casting Alloy 380	856
Aluminum Cube Ultra Purity	RM 1C
Aluminum 2-Ethylhexanoate	1075a

Name	SRM
Aluminum, Freezing Point Standard	44f
Aluminum, Magnetic Gram Susceptibility	763
Aluminum Oxide, Melting Point	742
Aluminum Rod Ultra Purity	RM 1R
Aluminum-26 Radioactivity Standard	4229
Americium-241 Alpha-Particle Standard	4904F
Americium-241 Gamma-ray Standard	4213
Ammonium Dihydrogen Phosphate	194
Angiotensin I (Human)	998
Anisic Acid	142
Anticonvulsant Drug Level Assay Standard	1599
Antiepilepsy Drug Level Assay Standard	900
Antimony-125-Tellurium-125m, Europium-154, Europium-155 Mixed-Radionuclide Point-Source Standard	4275B
Antimony-125-Tellurium-125m, Europium-154, Europium-155 Mixed-Radionuclide Solution Standard	4276B
A.O.H., 0.4C Spectrographic Steel Standard	413
Argillaceous Limestone	1C
Arsenic Trioxide Reductometric Standard	83d
Assay-Isotopic Standard for Potassium	985
Assay-Isotopic Standard for Rhenium	989
Assay-Isotopic Standard for Silicon	990
Assay-Isotopic Standard for Strontium	987
2% Austenite in Ferrite	488
5% Austenite in Ferrite	485a
15% Austenite in Ferrite	486
30% Austenite in Ferrite	487

Name	SRM	Name	SRM
Austenitic Stainless Steel, Thermal Conductivity and Electrical Resistivity	1460	Beryllium on Filter Media	2675
Austenitic Stainless Steel, Thermal Conductivity and Electrical Resistivity	1461	Besemer Steel (Simulated) 0.1% Carbon	8j
Austenitic Stainless Steel, Thermal Conductivity and Electrical Resistivity	1462	Bilirubin	916
Barium Crown Glass	713	Bis(1-phenyl-1, 3-butanediono) copper (II)	1080a
Barium Cyclohexanecbutyrate	1051b	Bis(1-phenyl-1, 3-butanediono) oxovanadium (IV)	1052b
Barium-133 Radioactivity Point-Source Standard	4241B	Black Porcelain Enamel for Directional Hemispherical Reflectance	2021
Barium-133 Radioactivity Standard	4251B	Black Porcelain Enamel for Directional Hemispherical Reflectance	2022
Basalt Rock	688	Blast Furnace Iron Standard (Chill Cast White)	1143a
Base Oil	1083	Blast Furnace Iron Standard (Chill Cast White)	1144a
Basic Electric Spectrographic Steel Standard	404a	B.O.H., 0.4C Spectrographic Steel Standard	417a
Basic Open-Hearth Steel, 0.1% Carbon	15g	Boric Acid	951
Basic Open-Hearth Steel, 0.1% Carbon	335	Boron-Doped Silicon Slices for Resistivity Measurements	1521
Basic Open-Hearth Steel, 0.1% Carbon	1228	Borosilicate Glass	93a
Basic Open-Hearth Steel, 0.2% Carbon	11h	Borosilicate Glass	623
Basic Open-Hearth Steel, 0.4% Carbon	12H	Borosilicate Glass	717
Basic Open-Hearth Steel, 0.5% Carbon	152A	Borosilicate Glass	1825
Basic Open-Hearth Steel, 0.8% Carbon	14f	Borosilicate Glass, Thermal Expansion	731
Basic Open-Hearth Steel, 1% Carbon (Disk)	1227	Bovine Liver	1577a
Basic Open-Hearth Steel, 1.1% Carbon	16f	Bovine Serum Albumin	926
Basic Open-Hearth Steel, 1.1% Carbon	337	Bovine Serum Albumin (7% Solution)	927
0.4C Basic Oxygen Furnace Steel	178	Branched Polyethylene	1476
Bauxite (Arkansas)	69b	Brewers Yeast	1569
Bauxite (Dominican)	697	Bright Copper Microhardness Standard	1894
Bauxite (Jamaican)	698	Bright Nickel Microhardness Standard	1895
Bauxite (Surinam)	696	Bromobenzoic Acid	2142
Benzene in Nitrogen	1805	Burnt Refractory	76a
Benzene in Nitrogen	1806	Burnt Refractory	77a
Benzene Permeation Device	1911	Burnt Refractory	78a
Benzoic Acid	140b	Cadmium Cyclohexanecbutyrate	1053a
Benzoic Acid	350a	Cadmium, Vapor Pressure	746
Benzoic Acid Calorimetric Standard	39i	Calcium Carbonate	915
Benzothiazyl Disulfide Rubber Compound	373f	Calcium 2-Ethylhexanoate	1074a
Beryllium-Copper Standard	1122	Calcium in Low-Alloy (Silicon) Steel	1254
Beryllium-Copper Standard	C1122	Calcium Molybdate	71
Beryllium-Copper Standard	C1123	Calibrated Glass Beads	1004
		Calibrated Glass Beads	1017a
		Calibrated Glass Beads	1018a
		Calibrated Glass Spheres	1003a
		Carbon Dioxide in Air	1670
		Carbon Dioxide in Air	1671
		Carbon Dioxide in Air	1672
		Carbon Dioxide in Nitrogen	1674b
		Carbon Dioxide in Nitrogen	1675b
		Carbon Dioxide in Nitrogen (Combustion Efficiency Gas Standard)	2619a
		Carbon Dioxide in Nitrogen (Combustion Efficiency Gas Standard)	2620a

Name	SRM
Carbon Dioxide in Nitrogen (Combustion Efficiency Gas Standard)	2621a
Carbon Dioxide in Nitrogen (Combustion Efficiency Gas Standard)	2622a
Carbon Dioxide in Nitrogen (Combustion Efficiency Gas Standard)	2623a
Carbon Dioxide in Nitrogen (Combustion Efficiency Gas Standard)	2624a
Carbon Dioxide in Nitrogen (Combustion Efficiency Gas Standard)	2625a
Carbon Dioxide in Nitrogen (Combustion Efficiency Gas Standard)	2626a
Carbon Dioxide in Nitrogen (Mobile Source Emission Gas Standard)	2632
Carbon Dioxide in Nitrogen (Mobile Source Emission Gas Standard)	2633
Carbon Monoxide in Air (Ambient Air Quality Gas Standard)	2612a
Carbon Monoxide in Air (Ambient Air Quality Gas Standard)	2613a
Carbon Monoxide in Air (Ambient Air Quality Gas Standard)	2614a
Carbon Monoxide in Nitrogen	1677c
Carbon Monoxide in Nitrogen	1678c
Carbon Monoxide in Nitrogen	1679c
Carbon Monoxide in Nitrogen	1680b
Carbon Monoxide in Nitrogen	1681b
Carbon Monoxide in Nitrogen (Mobile Source Emission Gas Standard)	2635
Carbon Monoxide in Nitrogen (Mobile Source Emission Gas Standard)	2636
Carbon Monoxide in Nitrogen (Mobile Source Emission Gas Standard)	2637
Carbon Monoxide in Nitrogen (Mobile Source Emission Gas Standard)	2638
Carbon Monoxide in Nitrogen (Mobile Source Emission Gas Standard)	2639
Carbon Monoxide in Nitrogen (Mobile Source Emission Gas Standard)	2640
Carbon Monoxide in Nitrogen (Mobile Source Emission Gas Standard)	2641
Carbon Monoxide in Nitrogen (Mobile Source Emission Gas Standard)	2642
Carbon-14 Radioactivity Standard	4245
Carbon-14 Radioactivity Standard	4246
Carbon Steel	1224
Carbon Steel, 0.6%	13g
Cast Iron	4k
Cast Iron	5L
Cast Iron	6g
Cast Iron	7G
Cast Iron Car Wheel	122h
Cast Steel 3	C1173
Cast Steel Standard	1138a
Cast Steel Standard	1139a

Name	SRM
Catalyst Package for Lubricant Oxidation	1817
Centerline Drawings for Optical Character Recognition, B Characters	1901
Centroid Color Chart	2106
Centroid Color Kit	2107
Cesium-137, Barium-137m Point-Source Radioactivity Standard	4200B
Cesium-137, Barium-137m Point-Source Radioactivity Standard	4207
Cesium-137 Burn-Up Standard	4233B
Cesium-134 Radioactivity Standard	4250B
Channel Black Rubber Compound	375g
Chlorine-36 Beta-ray Standard	4943
Chlorine-36 Radioactivity Standard	4422L
Chlorobenzoic Acid	2144
Chrome Refractory	103a
Chromium-Molybdenum-Aluminum Steel	106B
Chromium-Molybdenum Steel	36b
Chromium-Molybdenum Steel	133B
Chromium-Nickel-Molybdenum Steel	139b
Chromium-Nickel-Molybdenum Steel	1222
17Chromium-9 Nickel-0.2 Selenium Steel	339
Chromium-Nickel Spectrographic Steel Standard	408a
15Chromium-7 Nickel Steel	344
16Chromium-4 Nickel Steel	345
Chromium-51 Radioactivity Standard	4400L-F
Chromium Steel	163
Chromium-Tungsten Steel	155
Chromium-Vanadium Spectrographic Steel Standard	407a
Cholesterol	911a
Chrysotile Asbestos Fibers	1876
Citrus Leaves	1572
Clinical Laboratory Thermometer	934
Cobalt Cyclohexanecarboxylate	1055b
Cobalt-Molybdenum-Tungsten Steel	153A
Cobalt-57 Radioactivity Standard	4408L-C
Cobalt-60 Radioactivity Standard	4915D
Commercial Bronze Standard for Optical Emission and X-ray Spectroscopic Analysis	1115
Commercial Bronze Standard for Optical Emission and X-ray Spectroscopic Analysis	C1115
Commercial Bronze Standard for Optical Emission and X-ray Spectroscopic Analysis	1116

Name	SRM	Name	SRM
Commercial Bronze Standard for Optical Emission and X-ray Spectroscopic Analysis	C1116	Cupro-Nickel, 10% (CDA 706) High Purity	874
Commercial Bronze Standard for Optical Emission and X-ray Spectroscopic Analysis	1117	Cystine	143c
Commercial Bronze Standard for Optical Emission and X-ray Spectroscopic Analysis	C1117	Dextrose	41b
Common Lead Isotopic Standard	981	D-Glucose	917
Copper Concentrate	332	Dibutyltin Bis(2-ethylhexanoate)	1057b
Copper Heat Capacity Test Specimen	RM5	Didymium Glass Filter for Checking the Wavelength Scale of Spectrophotometers	2009
Copper-Nickel-Chromium Cast Iron	115A	Didymium Glass Filter for Checking the Wavelength Scale of Spectrophotometers	2010
Copper Ore, Mill Heads	330	Disodium Hydrogen Phosphate	186IIc
Copper Ore, Mill Tails	331	Disodium Hydrogen Phosphate	2186II
Copper-Thermal Expansion	736a	D-Mannitol	920
Copper, Secondary Freezing Point Standard	45d	Dolomitic Limestone	88a
Cortisol (Hydrocortisone)	921	Doped Platinum	681L1
Creatinine	914	Doped Platinum	681L2
Cr-Mo Low Alloy Steel	1270	Ductile Cast Iron	341
Cr-Mo Steel (ASTM A-213)	291	Electrical Residual Resistivity Ratio Standard	769
Cr-Mo (SAE 4140) Spectrographic Steel Standard	414	Electrolytic Iron	365
Cr-Mo (SAE 4150) Spectrographic Steel Standard	427	Electrolytic Iron	1265a
Cr-Mo (SAE X4130) Spectrographic Steel Standard	418a	Electrolytic Iron, Thermal Conductivity and Electrical Resistivity	1463
Cr-Ni-Mo Steel (AISI 8620)	293	Electrolytic Iron, Thermal Conductivity and Electrical Resistivity	1464
18Cr-10Ni Steel (AISI 304L)	101f	Electronic and Magnetic Alloy Standard	1159
Cr-V Steel (Modified)	363	Electronic and Magnetic Alloy Standard	1160
Cr-V Steel (Modified)	1263a	Enriched Boric Acid	952
Cr-V Steel (SAE 6150)	30f	Equal-Atom Lead Isotopic Standard	982
Crystalline Potassium Dichromate	935	Estuarine Sediment	1646
Crystalline Potassium Iodide, Heterochromatic Stray Radiant Energy Standard	2032	Europium-152 Point-Source Standard	4218E
Crystalline (Ruby) Electron Paramagnetic Resonance Absorption Intensity Standard	2601	Europium-152 Radioactivity Standard	4370B
Cupro-Nickel (CDA 706)	1275	Extra Dense Lead Glass	709
Cupro-Nickel (CDA 715)	1276	Fe-Cr-Ni Alloy Microprobe Standard	479a
Cupro-Nickel, 10% (CDA 706) Doped	875	Fe-Si Alloy Microprobe Standard	483
		Feldspar	70a
		Feldspar	99a
		Ferrochromium (Low Carbon)	196
		Ferrochromium Silicon	689
		Ferroniobium	340
		Ferrophosphorus	90
		Ferrosilicon	58a
		Ferrosilicon	59a
		Ferrosilicon (75% Si)	195
		First Surface Aluminum Mirror for Specular Reflectance	2003a
		First Surface Mirror, Gold on Glass	2008a



Name	SRM
Fission Track Glass Standard	961
Fission Track Glass Standard	962a
Fission Track Glass Standard	963a
Fission Track Glass Standard	964
Flint Clay	97a
Fluorobenzoic Acid	2143
Fluorspar	79a
Free-Cutting Brass	1103
Free-Cutting Brass	C1104
Freeze-Dried Urine	2670
Freeze-Dried Urine Certified for Fluoride	2671a
Freeze-Dried Urine Certified for Mercury	2672a
Fused Silica Thermal Expansion	739
Gadolinium-148 Alpha-Particle Standard	4907
Gallium Melting-Point Standard	1968
Gallium-67 Radioactivity Standard	4416L-D
Gas Furnace Black Rubber Compound	382a
Gasometric Set (1095-1099)	1089
Gasometric Standard for Unalloyed Zirconium	357
Gasometric Standard for Unalloyed Zirconium	358
Generator Columns for Polynuclear Aromatic Hydrocarbons	1644
Gilding Metal	1112
Gilding Metal	C1112
Gilding Metal	1113
Gilding Metal	C1113
Gilding Metal	1114
Gilding Metal	C1114
Glasses for Microchemical Analysis	1871
Glasses for Microchemical Analysis	1872
Glasses for Microchemical Analysis	1873
Glasses for Microchemical Analysis	1874
Glasses for Microchemical Analysis	1875
Glass Fibers for Microanalysis	RM 31
Glass Filter for Transmittance Measurement	2030
Glass Filters for Spectrophotometry	930D
Glass Fluorescence Source	477
Glass Sand	81a
Glass Sand	165a
Glass Spheres	1019a
Gold Coating on Glass Sealing Alloy	1398a
Gold Coating on Nickel	1379
Gold Coating on Nickel	1380
Gold Coating on Nickel	1399b
Gold-Copper Wires for Microprobe Analysis	482
Gold-195 Radioactivity Standard	4421L

Name	SRM
Gold-198 Radioactivity Standard	4405L-B
Gold-Silver Wires for Microprobe Analysis	481
Gold, Vapor Pressure	745
Gray Cast Iron	334
Halocarbons (in methanol) for Water Analysis	1639
High-Alloy Steel (A-743)	C1288
High-Alloy Steel (AISI 310 Mod.)	C1287
High-Alloy Steel, (AISI 414 Mod.)	C1289
High-Alloy White Cast	892
High-Alloy White Cast Iron	890
High-Alloy White Cast Iron	891
High-Carbon Ferrocromium	64c
High-Carbon Ferromanganese	68c
High-Carbon Steel (Modified)	364
High-Carbon Steel (Modified)	1264a
High-Grade Fluorspar	180
High-Nickel Steel	126c
High-Nickel Steel	1158
High-Purity Gold	685
High-Purity Platinum	680L1A
High-Purity Platinum	680L2A
High-Purity Platinum Thermoclement	1967
High-Purity Zinc	682
High-Silicon Steel	179
High-Silicon Steel	1134
High-Silicon Steel	1135
High-Silicon Steel (Calcium Bearing)	125b
High-Sulfur Steel	105
High-Sulfur Steel	129c
High-Sulfur Steel	1136
High Temperature Alloy A286	348
High Temperature Alloy M308	1197
High Temperature Alloy L605 and S816	S1199
High-Temperature Alloy	1206-2
High-Temperature Alloy	1207-1
High-Temperature Alloy	1207-2
High-Temperature Alloy	1208-1
High-Temperature Alloy	1208-2
Homogeneous River Sediment for Radioactivity Measurements	RM 45B
Human Liver, Environmental Radioactivity	4352
Human Lung, Environmental Radioactivity	4351
Human Serum	909

Name	SRM	Name	SRM
Hydrogen in Unalloyed Titanium	352b	Iron Ore (Sibley)	27f
Hydrogen in Unalloyed Titanium	1086	Iron Ore Concentrate (Canada)	690
Hydrogen in Unalloyed Titanium	1087	Iron-59 Radioactivity Standard	4411L-B
Hydrogen in Unalloyed Titanium	1088	Isobutylene-Isoprene (Butyl) Rubber	1495
Hydrogen-3 Radioactivity Standard	4361	Isobutylene-Isoprene (Butyl) Rubber	388L
Hydrogen-3 Radioactivity Standard	4926C	Isotopic Standard for Bromine	977
Hydrogen-3 Toluene Radioactivity Standard	4947	Isotopic Standard for Chlorine	975
4-Hydroxy-3-methoxy-DL-mandelic Acid (VMA)	925	Isotopic Standard for Chromium	979
ICTA High Temperature Set	GM 760	Isotopic Standard for Copper	976
Differential Thermal Analysis		Isotopic Standard for Magnesium	980
ICTA Low Temperature Set Differential Thermal Analysis	GM 757	Isotopic Standard for Silver	978
ICTA Mod Temperature Set Differential Thermal Analysis	GM 759	Krypton-85 Gaseous Radioactivity Standard	4308C
ICTA Mid Temperature Set Differential Thermal Analysis	GM 758	Krypton-85 Radioactivity Standard	4235
ICTA Polystyrene Differential Thermal Analysis	GM 754	Krypton-85 Radioactivity Standard	4935C
ICTA Thermogravimetry Set	GM 761	Lead-Barium Glass	89
Incoloy, 901 and Hastelloy X	S1198	Lead Base Bearing Metal	53c
Inconels, Alloy 600 (Chips)	864	Lead Base Bearing Metal	1132
Inconels, Alloy 600 (Solid)	1244	Lead Cyclohexanecarboxylate	1059c
Inconels, Alloy 625 (Chips)	865	Lead in Reference Fuel	1636a
Inconels, Alloy 625 (Solid)	1245	Lead in Reference Fuel	1637a
Incoloy, Alloy 800 (Chips)	866	Lead in Reference Fuel	1638a
Incoloy, Alloy 800 (Solid)	1246	Lead Nitrate	928
Incoloy, Alloy 825 (Chips)	867	Lead on Filter Media	2674
Incoloy, Alloy 825 (Solid)	1247	Lead-203 Radioactivity Standard	4420L
Indium-111 Radioactivity Standard	4417L-C	Lead, Secondary Freezing Point Standard	49c
Ingot Iron Spectrographic Steel Standard	420a	Lead-Silica Glass	1827
Intermediate Purity Selenium	726	Lead-Silica Glass (Viscosity)	711
Intermediate Purity Zinc	728	Lead-Silica Glass for dc Volume Resistivity	624
Iodine-123 Radioactivity Standard	4414L-C	Lead-Silica Glass for Dielectric Constant	774
Iodine-125 Radioactivity Standard	4407L-H	Lead 206 Spike Assay and Isotopic Solution Standard	991
Iodine-129 Radioactivity Standard	4949B	Leaded Tin Bronze Alloy	1035
Iodine-131 Radioactivity Standard	4401L-I	Light Sensitive Paper	700d
Iron Foil Mössbauer Standard	1541	Light Sensitive Paper	701d
Iron-55 Low-Energy Photon Standard	4260C	Light Sensitive Plastic Chip	703
Iron Metal (Clinical Standard)	937	Linear Polyethylene	1475
Iron Ore (Labrador)	692	Linear Polyethylene	1482
Iron Ore (Nimba)	693	Linear Polyethylene	1483
		Linear Polyethylene	1484
		Linerboard, Standard for Tape Adhesion Testing	1810
		Liquid Absorbance Standard for Ultraviolet and Visible Spectrophotometry	931c
		Lithium Carbonate	924
		Lithium Ore	181
		Lithium Ore	182
		Lithium Ore	183
		Low-Alloy Steel, (AISI 4130)	1225
		Low-Alloy Steel	1226
		Low-Alloy Steel (A242 Mod.)	C1285
		Low-Alloy Steel, AISI 4130	72g
		Low-Alloy Steel (AISI 1526, Modified)	1269
		Low-Alloy Steel (Hy 80)	1286

Name	SRM	Name	SRM
Low-Alloy Steel Set (661-665)	S668	Naval Brass Standards for Optical Emission and Spectroscopic Analysis	1108
Low-Carbon Silicon Steel	131c	Naval Brass Standards for Optical Emission and Spectroscopic Analysis	C1108
Low-Carbon Silicon Steel	1036		
Low-Carbon Stainless Steel (AISI 316L)	166c		
Magnesium-base Alloy	171		
Magnesium Cyclohexanecarboxylate	1061c	Neutral Glass	716
Magnesium Gluconate Dihydrate	929	Neutron Density Monitor Wire	953
Magnetic Coating on Magnetic Substrate (Nickel on Steel)	1365a	Nickel-Chromium Cast Iron	82b
Magnetic Coating on Magnetic Substrate (Nickel on Steel)	1366a	Nickel-Chromium-Molybdenum Cast Iron	107C
Magnetic Coating on Non-Magnetic Substrate (Nickel and Chromium on brass)	1367a	Nickel-Chromium Steel	32E
Magnetic Tape, High Density	6250	Nickel-Copper Alloy	882
Manganese Fluoride, Magnetic Gram Susceptibility	766	Nickel Cyclohexanecarboxylate	1065b
Manganese Ore	25d	Nickel Oxide, No. 1	671
Manganese-54 Point-Source Radioactivity Standard	4997E	Nickel Oxide, No. 2	672
Manganese-54 Radioactivity Standard	4257	Nickel Oxide, No. 3	673
Manganese Steel	100B	Nickel-63 Radioactivity Standard	4226
Manganese Cyclohexanecarboxylate	1062b	Nickel Silver (CDA 762)	879
Managing Steel	1156	Nickel Silver (CDA 770)	880
Metal on Quartz Filters for Spectrophotometry	2031	Nickel Spectrographic Steel Standard	409b
Metals on Filter Media	2676b	Nickel Sphere, Magnetic Moment	772
Methane in Air	1658a	Nickel Steel	33d
Methane in Air	1659a	Ni-Cr-Mo-V Steel	1173
Methane in Air	1660a	Nicotinic Acid	148
Medium Manganese Spectrographic Steel Standard	405a	Niobium-94 Gamma-ray Standard	4201B
Mercaptobenzothiazole	383a	Nitric Oxide in Nitrogen	1683b
Mercury, Freezing Point	743	Nitric Oxide in Nitrogen	1684b
Mercury-203 Radioactivity Standard	4418L	Nitric Oxide in Nitrogen	1685b
Mercury in Water, µg/mL	1641b	Nitric Oxide in Nitrogen	1686b
Mercury in Water, µg/mL	1642b	Nitric Oxide in Nitrogen	1687b
Microcopy Resolution Test Chart	1010a	Nitric Oxide in Nitrogen (Mobile Source Emission Gas Standard)	2627
Microprobe Standard – Cartridge Brass	478	Nitric Oxide in Nitrogen (Mobile Source Emission Gas Standard)	2628
Mineral Glasses for Microanalysis	470	Nitric Oxide in Nitrogen (Mobile Source Emission Gas Standard)	2629
Molybdenum Concentrate	333	Nitric Oxide in Nitrogen (Mobile Source Emission Gas Standard)	2630
Molybdenum, Heat Capacity	781	Nitric Oxide in Nitrogen (Mobile Source Emission Gas Standard)	2631
Molybdenum-99 Radioactivity Standard	4412L-H	Nitrogen Dioxide in Air (Stationary Source Emission Gas Standard)	2653
Molybdenum-Tungsten-Chromium-Vanadium Steel	134A	Nitrogen Dioxide in Air (Stationary Source Emission Gas Standard)	2654
Naval Brass Standards for Optical Emission and Spectroscopic Analysis	1106	Nitrogen Dioxide in Air (Stationary Source Emission Gas Standard)	2655
Naval Brass Standards for Optical Emission and Spectroscopic Analysis	C1106	Nitrogen Dioxide in Air (Stationary Source Emission Gas Standard)	2656
Naval Brass Standards for Optical Emission and Spectroscopic Analysis	1107	Nitrogen Dioxide Permeation Device	1629a
Naval Brass Standards for Optical Emission and Spectroscopic Analysis	C1107	4-Nitrophenol	938

Name	SRM	Name	SRM
Nodular Cast Iron	342a	Organics in Shale Oil	1580
Nominal One Micrometer Polystyrene Spheres	1690	Oxalic Acid	4990C
Non-Fat Powdered Milk	1549	Oxygen in Ferrous Materials	1090
Nonmagnetic Coating on Magnetic Substrate (Copper and Chromium on Steel)	1359	Ingot Iron	
Nonmagnetic Coating on Magnetic Substrate (Copper and Chromium on Steel)	1360	Oxygen in Ferrous Materials (Stainless Steel AISI 431)	1091
Nonmagnetic Coating on Magnetic Substrate (Copper and Chromium on Steel)	1361b	Oxygen in Ferrous Materials Vacuum Melted Steel	1092
Nonmagnetic Coating on Magnetic Substrate (Copper and Chromium on Steel)	1362a	Oxygen in Maraging Steel	1094
Nonmagnetic Coating on Magnetic Substrate (Copper and Chromium on Steel)	1363a	Oxygen in Nitrogen (Gas Standard)	2657
Nonmagnetic Coating on Magnetic Substrate (Copper and Chromium on Steel)	1364a	Oxygen in Nitrogen (Gas Standard)	2658
NPL GM Alpha Alumina	8005	Oxygen in Nitrogen (Gas Standard)	2659
NPL GM Alpha Alumina	8006	Oxygen in Titanium-Base Materials	355
NPL GM Alpha Alumina	8007	Oxygen in Valve Steel	1093
NPL GM Alpha Alumina	8008	Oyster Tissue	1566
NPL GM Graphitized Carbon Black	8001	Palladium, Magnetic Gram	765
NPL GM Graphitized Carbon Black	8002	Susceptibility	
NPL GM Melting Point Set	8000	Penetrant Test Block	1850
NPL GM Non-porous Silica	8003	Peruvian Soil, Environmental	4355
NPL GM Non-porous Silica	8004	Radioactivity	
N-tertiary Butyl-2-benzothiazolesulfenamide Rubber Compound	384d	Petroleum Crude Oil	1582
Obsidian Rock	278	Phosphate Rock (Florida)	120b
Octaphenylcyclotetrasiloxane	1066a	Phosphor Bronze (CDA 521)	871
Oil Furnace Black Rubber Compound	378b	Phosphor Bronze (CDA 544)	872
Opal Glass Powder	91	Phosphorized Copper, Cu VIII	C1251
Optical Emission and X-ray Spectroscopic Analysis	1102	Phosphorized Copper, Cu IX	C1252
Optical Microscope Linewidth Measurement Standard	474	Phosphorized Copper, Cu X	C1253
Optical Microscope Linewidth Measurement Standard	475	Phosphorus-32 Radioactivity Standard	4406L-G
Optical Microscope Linewidth Measurement Standard	476	Photographic Step Tablet	1008
		Fine Needles	1575
		Plastic Clay	98a
		Platinum, Magnetic Gram	764
		Susceptibility	
		Plutonium-238 Alpha-Particle Standard	4906B
		Plutonium-240 Alpha-Particle Emission-Rate Solution Standard	4338
		Plutonium-239 Alpha-Particle Solution Standard	4331
		Plutonium-242 Alpha-Particle Solution Standard	4334B
		Plutonium Isotopic Standard	946
		Plutonium Isotopic Standard	947
		Plutonium Isotopic Standard	948
		Plutonium Metal	949f
		Plutonium Metal (Standard Matrix Material)	945
		Plutonium-244 Spike Assay and Isotopic Standard	996
		Polychlorinated Biphenyls in Oil	1581
		Polycrystalline Alumina Elasticity Standard	718
		Polyester Plastic Film for Oxygen Gas Transmission	1470
		Polyisobutylene Solution in Cetane	1490
		Polystyrene	1478
		Polystyrene	1479
		Polystyrene (Broad Molecular Weight)	706
		Polystyrene (Narrow Molecular Weight)	705
		Polystyrene Spheres	1691
		Portland Cement (Black)	1880

Name	SRM	Name	SRM
Portland Cement (Blue)	635	Quartz on Filter Media	2679a
Portland Cement (Clear)	639	Quinine Sulfate Dihydrate	936
Portland Cement (Gold)	634	Radiogenic Lead Isotopic Standard	983
Portland Cement (Green)	638	Radium-226 Gamma-ray Standard	4956
Portland Cement (Pink)	637	Radium-226 Gamma-ray Standard	4957
Portland Cement (Red)	633	Radium-226 Gamma-ray Standard	4958
Portland Cement (White)	1881	Radium-226 Gamma-ray Standard	4959
Portland Cement (Yellow)	636	Radium-226 Gamma-ray Standard	4960
Portland Cement Fineness Standard	114n	Radium-226 Gamma-ray Standard	4961
Potassium Chloride	2202	Radium-226 Gamma-ray Standard	4962
Potassium Chloride (Clinical Standard)	918	Radium-226 Gamma-ray Standard	4963
Potassium Chloride (Primary Chemical)	999	Radium-226 Gamma-ray Standard	4964B
Potassium Chloride for Solution Calorimetry	1655	Radium Standard (Blank Solution)	4952B
Potassium Dichromate	136d	Radon-226 for Radon Analysis	4953C
Potassium Dihydrogen Phosphate	200	Red Brass	1109
Potassium Dihydrogen Phosphate	1861c	Red Brass	C1109
Potassium Dihydrogen Phosphate	2186f	Red Brass	1110
Potassium Erucate	1076	Red Brass	C1110
Potassium Feldspar	607	Red Brass	C1111
Potassium Fluoride	2203	Reduced Iron Oxide	691
Potassium Hydrogen Phthalate	185e	Reference Fuel Isooctane	1816a
Potassium Hydrogen Tartrate	188	Reference Fuel n-Heptane	1815a
Potassium Iodide with Attenuator	2033	Reflection Step Tablet	2061
Potassium Nitrate	193	Refractive Index Glass	1820
Potassium Tetroxalate	189	Refractive Index Silicone Liquids	1823
Powdered Lead Based Paint	1579	Refractive Index, Soda-Lime Glass	1822
Priority Pollutant Polynuclear Aromatic Hydrocarbons (in Acetonitrile)	1647	Relative Stress-Optical Coefficient Glass	708
Propane in Air	1665b	Resulfurized-Rephosphorized Steel	C1221
Propane in Air	1666b	Rice Flour	1568
Propane in Air	1667b	River Sediment	1645
Propane in Air	1668b	River Sediment, Environmental Radioactivity	4350B
Propane in Air	1669b	Rocky Flats Soil Number 1, Environmental Radioactivity	4353
Propane in Nitrogen (Mobile Source Emission Gas Standard)	2643	Rubidium Melting Point	1969
Propane in Nitrogen (Mobile Source Emission Gas Standard)	2644	Rutile Ore	670
Propane in Nitrogen (Mobile Source Emission Gas Standard)	2645	Scanning Electron Microscope Magnification Standard	484c
Propane in Nitrogen (Mobile Source Emission Gas Standard)	2646	Scanning Electron Microscope Performance Standard	2069
Propane in Nitrogen (Mobile Source Emission Gas Standard)	2647	Secondary Standard Flexible Disk Cartridge (Computer Amplitude Reference)	3210
Propane in Nitrogen (Mobile Source Emission Gas Standard)	2648	Secondary Standard Magnetic Tape	3200
Propane in Nitrogen (Mobile Source Emission Gas Standard)	2649	Secondary Standard Magnetic Tape Cassette	1600
Propane in Nitrogen (Mobile Source Emission Gas Standard)	2650	Secondary Standard Magnetic Tape Cartridge (Computer Amplitude Reference)	3216
Propane in Nitrogen and Oxygen (Mobile Source Emission Gas Standard)	2651	Second Surface Aluminum Mirror for Specular Reflectance	2023
Propane in Nitrogen and Oxygen (Mobile Source Emission Gas Standard)	2652		
Quartz Cuvette for Spectrophotometry	932		
Quartz for Hydrofluoric Acid Solution Calorimetry	1654		

Name	SRM	Name	SRM
Second Surface Aluminum Mirror for Specular Reflectance	2024	Soda-Lime Sheet Glass	1831
Second Surface Aluminum Mirror with Wedge for Specular Reflectance	2025	Soda-Lime Silica Glass	622
Selenium-Bearing Steel	1170b	Soda-Lime Silica Glass for Liquidus Temperature	710
Selenium-75 Radioactivity Standard	4409L-D	Sodium Bicarbonate	773
Sheet Brass	37E	Sodium Bicarbonate	191a
Silica Brick	198	Sodium Bicarbonate	2191
Silica Brick	199	Sodium Carbonate	192a
Silicon-Aluminum Alloy	87a	Sodium Carbonate	2192
Silicon Bronze	158A	Sodium Chloride	2201
Silicon Density Standard	1840	Sodium Chloride (Clinical Standard)	919
Silicon Density Standard	1841	Sodium Cyclohexanecarboxylate	1069b
Silicon Metal	57a	Sodium Oxalate Reductometric Standard	40h
Silicon Powder, Spacing Standard for X-ray Diffraction	640a	Sodium Pyruvate	910
Silicon Power Device Level Resistivity Standard	1522	Sodium Tetraborate Decahydrate (Borax)	187b
Silicon Resistivity Standard for Eddy Current Testers	1523	Solder	127b
Silver 2-Ethylhexanoate	1077a	Solder	1131
Silver-Gold Thermocouple Wire	733	Special Nuclear Container DOT 6M, 15 gal.	9940
Silver, Vapor Pressure	748	Special Nuclear Container, 55 gal.	9941
Sintered and Arc-Cast Tungsten, Thermal Conductivity and Electrical Resistivity	1465	Special Nuclear Container Type A, 10 gal.	9942
Sintered and Arc-Cast Tungsten, Thermal Conductivity and Electrical Resistivity	1466	Special Nuclear Container, Type A, 55 gal.	9943
Sintered and Arc-Cast Tungsten, Thermal Conductivity and Electrical Resistivity	1467	Special Nuclear Material Package	9910
Sintered and Arc-Cast Tungsten, Thermal Conductivity and Electrical Resistivity	1468	Spectrographic Ingot Iron and Low-Alloy Steel Standard (Rod)	461
Smoke Density Chamber Standard (Flaming Exposure Condition)	1007a	Spectrographic Ingot Iron and Low-Alloy Steel Standard (Rod)	462
Smoke Density Chamber Standard (Non-flaming Exposure Condition)	1006b	Spectrographic Ingot Iron and Low-Alloy Steel Standard (Rod)	463
Soda-Lime Container Glass	621	Spectrographic Ingot Iron and Low-Alloy Steel Standard (Rod)	464
Soda-Lime Flat Glass	620	Spectrographic Ingot Iron and Low-Alloy Steel Standard (Rod)	465
Soda-Lime Float Glass	1830	Spectrographic Ingot Iron and Low-Alloy Steel Standard (Rod)	466
Soda-Lime Glass	1826	Spectrographic Ingot Iron and Low-Alloy Steel Standard (Rod)	467
Soda-Lime Glass Powder	92	Spectrographic Ingot Iron and Low-Alloy Steel Standard (Rod)	468
		Spectrographic Ingot Iron and Low-Alloy Steel Standard (Rod)	1166
		Spectrographic Stainless Steel Standard	442
		Spectrographic Stainless Steel Standard	443
		Spectrographic Stainless Steel Standard	444
		Spectrographic Stainless Steel Standard (Disc)	D849
		Spectrographic Stainless Steel Standard (Disc)	D850
		Spectrographic Stainless Steel Standard (Group II)	445

Name	SRM	Name	SRM
Spectrographic Stainless Steel Standard (Group II)	446	Spectroscopic Titanium-Base Standard	644
Spectrographic Stainless Steel Standard (Group II)	447	Spectroscopic Titanium-Base Standard	645
Spectrographic Stainless Steel Standard (Group II)	448	Spectroscopic Titanium-Base Standard	646
Spectrographic Stainless Steel Standard (Group II)	449	Spheroidized Iron Carbide in Ferrite	493
Spectrographic Stainless Steel Standard (Group II)	450	Spreading Resistance Calibration (100) n-Type Silicon	2529
Spectrographic Stainless Steel Standard (Group II)	849	Spreading Resistance Calibration (100) p-Type Silicon	2528
Spectrographic Stainless Steel Standard (Rod)	850	Spreading Resistance Calibration (111) n-Type Silicon	2527
Spectrographic Steel Standard (Disc)	D803a	Spreading Resistance Calibration (111) p-Type Silicon	2526
Spectrographic Steel Standard (Disc)	D807a	Stabilized Wine	1590
Spectrographic Steel Standard (Rod)	803a	Stainless Steel	121d
Spectrographic Steel Standard (Rod)	804a	Stainless Steel	123c
Spectrographic Steel Standard (Rod)	805a	Stainless Steel	160b
Spectrographic Steel Standard (Rod)	807a	Stainless Steel (AISI 446)	367
Spectrographic Steel Standard (Rod)	808a	Stainless Steel (AISI 446)	1267
Spectrographic Steel Standard (Rod)	809a	Stainless Steel, 13% Chromium	73c
Spectrographic Steel Standard (Rod)	817b	Stainless Steel, Cr-Ni	C1151
Spectrographic Steel Standard (Rod)	820a	Stainless Steel, Cr-Ni	1151a
Spectrographic Steel Standard (Rod)	821	Stainless Steel, Cr-Ni	C1152
Spectrographic Steel Standard (Rod)	827	Stainless Steel, Cr-Ni	1152a
Spectrographic Tool Steel Standard	436	Stainless Steel, Cr-Ni	C1153
Spectrographic Tool Steel Standard	437	Stainless Steel, Cr-Ni	1153a
Spectrographic Tool Steel Standard	438	Stainless Steel, Cr-Ni-Mo	C1154
Spectrographic Tool Steel Standard	439	Stainless Steel, Cr-Ni-Nb	1154a
Spectrographic Tool Steel Standard	440	Stainless Steel, Cr-Ni-Ti	1155
Spectrographic Tool Steel Standard	441	Stainless Steel for Pitting or Crevice Corrosion	1172
Spectrographic Tool Steel Standard	837	Stainless Steel Thermal Expansion	1171
Spectrographic Tool Steel Standard	840	Stearic Acid Rubber Compound	1890
Spectrographic Tool Steel Standard	D837	Steel (AISI 1211)	738
Spectrographic Tool Steel Standard	D840	Steel (Lead-Bearing)	372h
Spectrographic Tool Steel Standard	D841	Strontium Cyclohexanecarboxylate	368
Spectrographic Zinc-Base Die-Casting Alloy A	625	Strontium-85 Radioactivity Standard	1169b
Spectrographic Zinc-Base Die-Casting Alloy B	626	Strontium-89 Radioactivity Standard	1070a
Spectrographic Zinc-Base Die-Casting Alloy C	627	Styrene-butadiene Rubber (Type 1500)	4403L-B
Spectrographic Zinc-Base Die-Casting Alloy D	628	Succinonitrile Freezing Point	4945D
Spectrographic Zinc-Base Die-Casting Alloy E	629	Sucrose	386h
Spectrographic Zinc-Base Die-Casting Alloy F	630	Sulfate and Nitrate on Filter Media	1970
Spectroscopic Titanium-Base Standard	631	Sulfur Dioxide in Nitrogen	17c
Spectroscopic Titanium-Base Standard	641	Sulfur Dioxide in Nitrogen	2673
Spectroscopic Titanium-Base Standard	642	Sulfur Dioxide in Nitrogen	1661a
Spectroscopic Titanium-Base Standard	643	Sulfur Dioxide in Nitrogen	1662a
		Sulfur Dioxide in Nitrogen	1663a
		Sulfur Dioxide in Nitrogen	1664a
		Sulfur Dioxide in Nitrogen	1693
		Sulfur Dioxide in Nitrogen	1694



Name	SRM	Name	SRM
Sulfur Dioxide in Nitrogen	1696	Titanium-Base Alloy (Unalloyed)	650
Sulfur Dioxide Permeation Tube (2 cm tube)	1627	Titanium-Base Alloy (Unalloyed)	651
Sulfur Dioxide Permeation Tube (5 cm tube)	1626	Titanium-Base Alloy (Unalloyed)	652
Sulfur Dioxide Permeation Tube (10 cm tube)	1625	Titanium Dioxide	154b
Sulfur in Coal	2682	Toluene	211c
Sulfur in Coal	2683	Tomato Leaves	1573
Sulfur in Coal	2684	Tool Steel (AISI M2)	132b
Sulfur in Coal	2685	Tool Steel (AISI M2)	1157
Sulfur in Residual Fuel Oil	1619	Tool Steel Abrasive Wear Standard	1857
Sulfur in Residual Fuel Oil	1620a	Tracealloy (Nickel-Base High-Temperature Alloy)	897
Sulfur in Residual Fuel Oil	1621b	Tracealloy (Nickel-Base High-Temperature Alloy)	898
Sulfur in Residual Fuel Oil	1622b	Tracealloy (Nickel-Base High-Temperature Alloy)	899
Sulfur in Residual Fuel Oil	1623a	Trace Elements in a Glass Matrix	610
Sulfur Rubber Compound	1624a	Trace Elements in a Glass Matrix	611
Superconductive Thermometric Fixed Point Device	767a	Trace Elements in a Glass Matrix	612
Superconductive Thermometric Fixed Point Device	768	Trace Elements in a Glass Matrix	613
Surface Flammability Standard	1002c	Trace Elements in a Glass Matrix	614
Synthetic Sapphire	720	Trace Elements in a Glass Matrix	615
Technetium-99 Radioactivity Standard	4288	Trace Elements in a Glass Matrix	616
Technetium-99m Radioactivity Standard	4410H-1	Trace Elements in a Glass Matrix	617
Tetrachloroethylene in Nitrogen	1808	Trace Elements in Coal (Bituminous)	1632a
Thallium-201 Radioactivity Standard	4404L-F	Trace Elements in Coal (Sub-bituminous)	1635
Thermal Resistance, Fibrous Glass Batt	1451	Trace Elements in Coal Fly Ash	1633a
Thermal Resistance, Fibrous Glass Board	1450b	Trace Elements in Fuel Oil	1634a
Thorium-228, Thallium-208 Gamma-ray Point-Source Standard	4206C	Trace Elements in Water	1643a
Tin-Base Bearing Metal	54D	Trace Mercury in Coal	1630
Tin, Freezing Point	741	2,2,4-Trimethylpentane	217c
Tin-113-Indium-113m Radioactivity Standard	4402L-C	Tripalmitin	1595
Tin-121m Point-Source Gamma-ray Emission-Rate Standard	4264B	Tris, Basimetric	723a
Tin, Secondary Freezing Point Standard	42g	Tris, for Solution Calorimetry	724a
Titanium Alloy	654a	Tris(hydroxymethyl)aminomethane	922
Titanium-Base Alloy	173b	Tris(hydroxymethyl)aminomethane hydrochloride	923
Titanium-Base Alloy	176	Tris(1-phenyl-1, 3-butanediono) Chromium (III)	1078b
		Tris(1-phenyl-1, 3-butanediono) Iron (III)	1079b
		Triphenyl Phosphate	1071b
		Tungsten Carbide	276a
		Tungsten-Chromium-Vanadium Steel	50c
		Tungsten Concentrate	277
		Tungsten, Heat Capacity	782
		Tungsten-20% Molybdenum Alloy	480
		Electron Microprobe Standard	
		Tungsten Thermal Expansion	737
		Unalloyed Copper	1034
		Unalloyed Copper, Cu "O"	393
		Unalloyed Copper, Cu IV	457
		Unalloyed Copper, Cu XI	454
		Unalloyed Copper, Cu I (Chip)	394
		Unalloyed Copper, Cu II (Chip)	395
		Unalloyed Copper, Cu III (Chip)	396
		Unalloyed Copper, Cu V (Chip)	398
		Unalloyed Copper, Cu VI (Chip)	399
		Unalloyed Copper, Cu VII (Chip)	400
		Unalloyed Copper, Cu I (Rod)	494

Name	SRM	Name	SRM
Unalloyed Copper, Cu II (Rod)	495	Wear-Metals in Lubricating Oil	1085
Unalloyed Copper, Cu III (Rod)	496	(300 ppm)	
Unalloyed Copper, Cu V (Rod)	498	Wheat Flour	1567
Unalloyed Copper, Cu VI (Rod)	499	White Cast Iron	338
Unalloyed Copper, Cu VII (Rod)	500	White Cast Iron (Disc)	1145
Unalloyed Titanium	354	White Cast Iron (Disc)	1146
Uranium Isotopic Standard (Nominally depleted to 0.02%)	U-0002	White Cast Iron (Disc)	1150
Uranium Isotopic Standard	U-005a	White Ceramic Tile for Directional Hemispherical Reflectance	2019b
Uranium Isotopic Standard	U-010	White Ceramic Tile for Directional Hemispherical Reflectance	2020
(Nominally 1% Enriched)		White Iron	3d
Uranium Isotopic Standard	U-015	White Opal Glass Diffuse Spectral Reflectance Standard for the Visible Spectrum	2015
(Nominally 1.5% Enriched)		Xenon-127 Gaseous Radioactivity Standard	4309G
Uranium Isotopic Standard	U-020	Xenon-133 Gaseous Radioactivity Standard	4307I
Uranium Isotopic Standard	U-030a	Xenon-133 Gaseous Radioactivity Standard	4415L-I
Uranium Isotopic Standard	U-050	Xenon-133, Xenon-137, Krypton-85 Mixed Gaseous Radioactivity Standard	4310B
(Nominally 5% Enriched)		X-ray Film Step Tablet	1001
Uranium Isotopic Standard	U-100	X-ray Powder Diffraction Intensity Standard	674
(Nominally 10% Enriched)		X-ray Powder Diffraction (Mica) Low 2 Theta	675
Uranium Isotopic Standard	U-150	Ytterbium-169 Radioactivity Standard	4419L-B
(Nominally 15% Enriched)		Zinc-Base Alloy (Die Casting)	94c
Uranium Isotopic Standard	U-200	Zinc Concentrates	113a
(Nominally 20% Enriched)		Zinc Concentrates	329
Uranium Isotopic Standard	U-350	Zinc Cyclohexanecarboxylate	1073b
(Nominally 35% Enriched)		Zinc, Freezing Point	740
Uranium Isotopic Standard	U-500	Zinc, Freezing Point Standard	43b
(Nominally 50% Enriched)		Zinc Metal	683
Uranium Isotopic Standard	U-750	Zinc Oxide Rubber Compound	370e
(Nominally 75% Enriched)		Zircaloy-2	360a
Uranium Isotopic Standard	U-800	Zircaloy-4 Metal	1237
(Nominally 80% Enriched)		Zircaloy-4 Metal	1238
Uranium Isotopic Standard	U-850	Zircaloy-4 Metal	1239
(Nominally 85% Enriched)		Zirconium-Barium Chromate Formulation for Heat-Source Powder Calorimetry	1651
Uranium Isotopic Standard	U-900	Zirconium-Barium Chromate Formulation for Heat-Source Powder Calorimetry	1652
(Nominally 90% Enriched)		Zirconium-Barium Chromate Formulation for Heat-Source Powder Calorimetry	1653
Uranium Isotopic Standard	U-930	Zirconium Metal	1234
(Nominally 93% Enriched)		Zirconium Metal	1235
Uranium Isotopic Standard	U-970	Zirconium Metal	1236
(Nominally 97% Enriched)			
Uranium Metal	960		
Uranium Oxide	950b		
Uranium Oxide	969		
Uranium-233 Spike Assay and Isotopic Solution Standard	995		
Urban Dust/Organics	1649		
Urban Particulate Matter	1648		
Urea	912a		
Urea	2141		
Urea	2152		
Uric Acid	913		
Vanadium and Nickel in Residual Fuel Oil	1618		
Vanadium in Crude Oil	8505		
Vanadium-49 Low-Energy Photon Standard	4266		
Waspaloy	349		
Wear-Metals in Lubricating Oil (100 ppm)	1084		

Appendix II. Certificates for Coal, Ore, Mineral, Rock, and Refractory  
Standards (listed in numerical order).

U.S. Department of Commerce  
Juanita M. Kreps  
Secretary

National Bureau of Standards  
Ernest Ambler, Director

# National Bureau of Standards

## Certificate of Analysis

### Standard Reference Material 1c

### Argillaceous Limestone

(In Cooperation with the American Society for Testing and Materials)

(All analyses are based on samples dried 2 hours at 110°C)

Constituent	SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	MnO	CaO	SrO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	Loss on Ignition
Certified Value, % by wt.	6.84	0.55	1.30	0.07	0.04	0.025	50.3	0.030	0.42	0.02	0.28	39.9
Estimated Uncertainty	0.08	0.03	0.03	0.01	0.01	0.005	0.3	0.005	0.04	0.01	0.01	0.1
Method <sup>a</sup>	Atomic Absorption	Atomic Absorption	Atomic Absorption	Atomic Absorption	Photometric	Atomic Absorption		Atomic Absorption	Atomic Absorption	Atomic Absorption	Atomic Absorption	
Labs												
A	<sup>a</sup> 6.82	<sup>b</sup> 0.53	<sup>b</sup> 1.33	<sup>c</sup> 0.07	<sup>d</sup> 0.04	<sup>e</sup> 0.03	<sup>f</sup> 50.40	---	<sup>f</sup> 0.45	<sup>g</sup> 0.03	<sup>g</sup> 0.28	39.93
B	<sup>h</sup> 6.77	<sup>i</sup> .61	<sup>j</sup> 1.31	<sup>c</sup> .07	<sup>d</sup> .03	<sup>e</sup> .02	<sup>k</sup> 50.19	---	<sup>i</sup> .54	---	<sup>g</sup> .29	39.80
C	<sup>m</sup> 6.82 <sup>n</sup> 6.77 <sup>h</sup> 6.80	.55	1.27	<sup>c</sup> .06	<sup>d</sup> .05	.02	<sup>n</sup> 50.18 <sup>m</sup> 50.56 <sup>o</sup> 50.20	0.03	<sup>m</sup> .38	.02	<sup>m</sup> .28 <sup>o</sup> .29	39.82 39.85
D	<sup>h</sup> 6.92	<sup>i</sup> .55	<sup>j</sup> 1.30	<sup>o</sup> .066	<sup>d</sup> .038	.022	50.18	.030	45	<sup>g</sup> .02	<sup>g</sup> .30	39.90
E	6.92	.57	1.29	<sup>m</sup> .066	<sup>d</sup> .039	.021	<sup>k</sup> 50.57	.031	41	.028	.27	39.87
F	6.76	.57	---	.08	.04	.027	50.52	.03	42	.02	---	39.97
G	<sup>p</sup> 6.91	.54	1.35	.07	<sup>d</sup> .04	.027	<sup>k</sup> 50.20	.03	42	.02	.28	39.89
H	---	---	---	---	---	.022	<sup>q</sup> 50.58 <sup>n</sup> 49.96	.034	.38	<sup>g</sup> .025	<sup>g</sup> .30	---

- The certified value listed for a constituent is the present best estimate of the "true" value based on results of the cooperative analytical program for certification.
- The estimated uncertainty of the "true" value is based on judgment and represents an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material variability for samples of 0.5 g or more. (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determination.)
- Detailed descriptions of many of the methods of analysis employed in the certification program for this SRM may be found in Part 13, Annual Book of ASTM Standards. They are also available as separate reprints, C25 and C114, from ASTM headquarters. ASTM Standard Technical Publication No. 395 also describes methods of analysis used in this certification work.

<sup>a</sup> Silicomolybdate photometric method.

<sup>b</sup> Ferron (8-hydroxy-7-oxo-5-quinolinesulfonic acid)

photometric method.

<sup>c</sup> Tiron (disodium 1,2-dihydroxybenzene-3,5-disulfonate)

photometric method.

<sup>d</sup> Molybdenum blue photometric method.

<sup>e</sup> Peroxydisulfate photometric method.

<sup>f</sup> EDTA titration.

<sup>g</sup> Flame emission spectrometry.

<sup>h</sup> Dehydration with HCl.

<sup>i</sup> SnCl<sub>2</sub> reduction-K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> titration.

<sup>j</sup> By difference between total NH<sub>4</sub>OH group and oxides of iron, phosphorus, and titanium.

<sup>k</sup> Calcium precipitated as oxalate and titrated with standard KMnO<sub>4</sub>.

<sup>l</sup> Magnesium determined gravimetrically as Mg<sub>2</sub>P<sub>2</sub>O<sub>7</sub>.

<sup>m</sup> X-ray fluorescence spectrometry.

<sup>n</sup> Atomic absorption spectrometry.

<sup>o</sup> H<sub>2</sub>O<sub>2</sub> photometric.

<sup>p</sup> Dehydration with HClO<sub>4</sub>.

Washington, D. C. 20234  
December 14, 1978

J. Paul Cali, Chief  
Office of Standard Reference Materials

(over)

#### PLANNING, PREPARATION, TESTING, AND ANALYSIS:

The material for this SRM was provided by Lone Star Industries, Inc., Cement and Construction Materials Group, Houston, Texas, through the courtesy of C. W. Moore.

At NBS, the material was ground, sieved and thoroughly blended.

Chemical analyses for certification were performed in the following laboratories:

Atlantic Cement Co., Inc., Ravena, N.Y., F. J. Hogan and W. Twiss.

California Portland Cement Co., Colton, Calif., P. Hawkins and N. Norton.

Ideal Basic Industries, Cement Division, Ft. Collins, Colo., J. W. Yule.

Lone Star Industries, Inc., Cement and Construction Materials Group, Houston, Texas, C. W. Moore, L. S. Scheline, and I. Z. Somcio.

Martin Marietta Laboratories, Baltimore, Md., E. H. Scott.

National Bureau of Standards, Center for Analytical Chemistry, Washington, D.C., T. C. Rains, M. B. Blackburn, T. J. Brady, J. D. Messman, and T. A. Rush, and by R. K. Bell, Assistant Research Associate, ASTM-NBS Research Associate Program.

Portland Cement Association, Skokie, Ill., W. F. Mivelaz, R. F. Crow, E. LaBonde, A. G. Mateos, C. P. Palmiano, and H. Seiler.

Universal Atlas Cement, Division of United States Steel Corp., Gary, Ind., Z. T. Jugovic.

The overall direction and coordination of the technical measurements leading to certification were performed by J. I. Shultz, Research Associate, ASTM-NBS Research Associate Program.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by W. P. Reed.

# National Bureau of Standards Certificate of Analysis

## Standard Reference Material 27f

### IRON ORE (Sibley)

(In Cooperation with the American Society for Testing and Materials)

This material is in the form of fine powder, for use in checking chemical methods of analysis and in calibration with instrumental methods of analysis.

(Results based on sample dried for one hour at 105 °C.)

Constituent	Total Fe	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	P	S	TiO <sub>2</sub>	MnO	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O
Certified <sup>1</sup> Value % by wt.	65.97	4.17	0.82	0.041	0.005	0.019	0.011	0.039	0.019	0.012	* 0.008
Estimated <sup>2</sup> Uncertainty	0.05	0.03	0.03	0.001	≤0.001	0.002	0.002	0.003	0.004	0.003	0.002
Method <sup>3</sup>	SnCl <sub>2</sub> K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub>	HClO <sub>4</sub> Dehydration	Atomic Absorption	Photometric	Combustion- Titration	Photometric	Atomic Absorption	Atomic Absorption	Atomic Absorption	Atomic Absorption	Atomic Absorption
Labs	SnCl <sub>2</sub> K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub>	HClO <sub>4</sub> Dehydration	Atomic Absorption	Photometric	Combustion- Titration	Photometric	Atomic Absorption	Atomic Absorption	Atomic Absorption	Atomic Absorption	Atomic Absorption
A	66.01	<sup>a</sup> 4.12	0.78	<sup>b</sup> 0.042	0.005	<sup>c</sup> 0.020	0.009	0.042	0.02	<sup>d</sup> 0.012	<sup>d</sup> 0.006
B	<sup>e</sup> 65.98	4.18	.83	.042	.006	<sup>f</sup> .018	<sup>g</sup> .014 <sup>g</sup> .012	<sup>g</sup> .041 <sup>g</sup> .042	<sup>g</sup> .013 <sup>g</sup> .015	<sup>d</sup> 0.012 <sup>g</sup> .010	<sup>d</sup> .011 <sup>g</sup> .009
C	65.93	<sup>h</sup> 4.18	.87 <sup>i</sup> .84	.039	<sup>j</sup> .005	<sup>k</sup> .019	<sup>l</sup> .010 <sup>l</sup> .011	<sup>d</sup> .039	.018	<sup>d</sup> .009	<sup>d</sup> .008
D	65.92	4.14	<sup>m</sup> .82	.041	.006	.02	.006	.036	.023	<sup>d</sup> 0.016	<sup>d</sup> .008
E	<sup>n</sup> 65.96	4.17	.79	<sup>b</sup> .040	<sup>j</sup> .005	<sup>o</sup> .021	<sup>p</sup> .011	.035	.021	.010	.009
F	<sup>n</sup> 66.04	-	-	<sup>b</sup> .042	-	<sup>q</sup> .017	.011	-	.027	.015	.007
G	<sup>o</sup> 65.96	4.20	<sup>r</sup> .82 <sup>r</sup> .81	.040	<sup>j</sup> .005	<sup>f</sup> .018	.011 <sup>l</sup> .013	.035	.018	.009	.007

- The certified value listed for a constituent is the *present best estimate* of the "true" value based on results of the cooperative analytical program for certification.
  - Estimated uncertainty includes method imprecision, bias among methods, and material variability for samples 0.5 g or more.
  - A detailed description of many of the methods of analysis employed in the certification program for this SRM may be found in Part 12, Chemical Analysis of Metals and Metal Bearing Ores, Annual Book of ASTM Standards.
- NOTE: Laboratory C reported a value of 0.002 percent ZrO<sub>2</sub> by the pyrocatechol photometric method.

<sup>a</sup> Sample fused in Na<sub>2</sub>O<sub>2</sub>.

<sup>b</sup> Alkali-molybdate method.

<sup>c</sup> X-ray spectrometric method.

<sup>d</sup> Flame emission spectrometry.

<sup>e</sup> H<sub>2</sub>S reduction-K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> titration.

<sup>f</sup> Chromotropic acid photometric.

<sup>g</sup> Spectrographic method.

<sup>h</sup> Sample dissolved in HCl, silica removed by double dehydration with HCl. Ignited silica treated with H<sub>2</sub>SO<sub>4</sub> and HF.

<sup>i</sup> Aluminum separated by anion-exchange and determined by colorimetric titration using 1,2-diaminocyclohexanetetracetic acid and back titration with standard zinc solution.

<sup>j</sup> Combustion-chromatographic

<sup>k</sup> Titanium separated by anion-exchange and determined photometrically with diantipyryl methane.

<sup>l</sup> Photometric

<sup>m</sup> Aluminum separated by anion-exchange and determined gravimetrically with phenylhydrazine.

<sup>n</sup> SnCl<sub>2</sub> reduction - KMnO<sub>4</sub> titration.

<sup>o</sup> H<sub>2</sub>O<sub>2</sub> photometric.

<sup>p</sup> Peroxydisulfate-arsenic.

<sup>q</sup> Atomic absorption spectrometry.

<sup>r</sup> Mercury cathode-NH<sub>4</sub>OH-Cupferron-AlPO<sub>4</sub>.

Washington, D.C. 20234  
May 31, 1977

J. Paul Cali, Chief  
Office of Standard Reference Materials

(Over)

PLANNING, PREPARATION, TESTING, ANALYSIS: The iron ore material for this SRM was provided to NBS by the United States Steel Corporation, Pittsburgh, Pa., through the courtesy of R. H. Colin.

The "as received" material was crushed, dry ground, and sieved under contract with the Colorado School of Mines Research Institute, Golden, Colorado, under direction of M. G. Pattengill and H. O. VanMale. The final product passed a 150 mesh (105 $\mu$ m) sieve, with about 50% passing a 200 mesh (74 $\mu$ m) sieve.

At NBS the material was sieved and thoroughly blended. Homogeneity testing of selected samples representative of the final lot was performed at NBS by R. K. Bell, Assistant Research Associate, ASTM/NBS Research Associate Program. The observed range of values is as follows:

Constituent	Range, %	No. of determinations	Sample size, grams
Fe	$\pm 0.05$	16	0.5
SiO <sub>2</sub>	$\leq \pm 0.02$	4	1.0
P	$\leq \pm 0.001$	4	0.5

It is concluded that the material variability is within the method imprecision.

Chemical analyses for certification were performed in the following laboratories:

Alan Wood Steel Company, Conshohocken, Pa., V. J. Mercaldo.

Andrew S. McCreath and Son, Inc., Harrisburg, Pa., R. F. Lippi.

Booth, Garrett and Blair, Inc., Ambler, Pa., J. H. Ormsbee.

Ledoux and Company, Teaneck, N. J., S. Kallman.

National Bureau of Standards, Analytical Chemistry Division, Washington, D.C. by T. C. Rains and S. A. Wicks, and by R. K. Bell, ASTM Assistant Research Associate.

United States Steel Corporation, Research Laboratory, Monroeville, Pa., by J. D. Selvaggio, D. G. Cunningham, J. DiNardi, J. B. Ferons, A. V. Fioravanti, J. E. Friedline, J. R. Lucas, II, K. G. Mikos, C. W. Ponsonby, D. Shafferman, and R. J. Wargo.

Weirton Steel Division, Weirton, W. Va., R. L. Zickefoose.

The overall direction and coordination of the technical measurements leading to certification were performed jointly by R. E. Michaelis, Office of Standard Reference Materials, and by J. I. Shultz, Research Associate, ASTM/NBS Research Associate Program.

The technical and support aspects involved in the preparation, certification and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by W. P. Reed.

U.S. Department of Commerce  
Juanita M. Kreps  
Secretary

National Bureau of Standards  
Ernest Ambler, Director

# National Bureau of Standards

## Certificate of Analysis

### Standard Reference Material 69b

#### Bauxite (Arkansas)

(In Cooperation with the American Society for Testing and Materials)

(All analyses are based on samples dried 2 hours at 140 °C)

This material is in the form of fine powder (<0.08 mm) for use in checking chemical and instrumental methods of analyses.

Constituent	Certified Value <sup>1</sup> Percent, by weight	Estimated Uncertainty <sup>2</sup>
Al <sub>2</sub> O <sub>3</sub>	48.8	0.2
Fe <sub>2</sub> O <sub>3</sub>	7.14	.12
SiO <sub>2</sub>	13.43	.10
TiO <sub>2</sub>	1.90	.05
ZrO <sub>2</sub>	0.29	.07
P <sub>2</sub> O <sub>5</sub>	.118	.004
V <sub>2</sub> O <sub>5</sub>	.028	.003
Cr <sub>2</sub> O <sub>3</sub>	.011	.002
CaO	.13	.02
MgO	.085	.008
MnO	.110	.005
ZnO	.0035	.0005
K <sub>2</sub> O	.068	.009
SO <sub>3</sub>	.63	.02
Loss on Ignition <sup>3</sup>	27.2	.2

<sup>1</sup>The certified value listed for a constituent is the *present best estimate* of the "true" value.

<sup>2</sup>The estimated uncertainty listed for a constituent is based on judgment and represents an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material variability for samples 1.0 g or more. (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determination of most constituents.)

<sup>3</sup>Determined by igniting to constant weight at 1050 °C.

Washington, D.C. 20234  
August 24, 1979

George A. Uriano, Chief  
Office of Standard Reference Materials

(over)



#### ADDITIONAL INFORMATION ON THE COMPOSITION

Elements other than those certified may be present in this material as indicated below. These are not certified but are given as additional information on the composition.

<u>Constituent</u>	<u>Concentration, Percent by weight</u>	<u>Constituent</u>	<u>Concentration, Percent by weight</u>
BaO	(0.008)	Co	(0.0001)
Na <sub>2</sub> O	(0.025)	Hf	(0.0063)
Ce	(0.024)	Sc	(0.0008)

The mineralogical composition of SRM 69b was determined by x-ray diffraction studies at the Geological Survey, U.S. Department of the Interior, Reston, Va., (J. W. Hosterman) to be 30% kaolinite, 60% gibbsite, and 10% siderite. These results are semiquantitative (to the nearest 5%).

#### PLANNING, PREPARATION, TESTING, ANALYSIS:

The mine run material for this SRM was provided by the Aluminum Company of America, Bauxite, Arkansas, through the courtesy of T.J. Forbes and by the Alcoa Technical Center, Pittsburgh, Pa., courtesy of H.B. Hartman. It was processed (crushed, ground, sieved, and mixed) at the Colorado School of Mines Research Institute under a contract with the National Bureau of Standards.

Homogeneity testing was performed at NBS by J.S. Maples and T.E. Gills.

Cooperative analyses for certification were performed in the following laboratories:

Aluminum Company of America, Alcoa Center, Pa., R. C. Obbink.  
 Aluminum Company of Canada, Ltd., Arvida Research Center, Arvida, Quebec, Canada, L. Girolami.  
 Andrew S. McCreath & Son, Inc., Harrisburg, Pa., F. A. Pennington, Jr., R. F. Eakin, and S. L. Miller.  
 General Refractories Co., U.S. Refractories Division, Research Center, Baltimore, Md., S. Banerjee.  
 Geological Survey, U.S. Department of the Interior, Reston, Va., H. J. Rose, Jr., and J. W. Hosterman.  
 Kaiser Aluminum and Chemical Corp., Center for Technology, Pleasanton, Calif., H. J. Seim, A. E. McLaughlin, D. F. G. Marten, A. Kermaninejad, R. C. Kinne, J. R. Skarset, J. Boruk, and U. Vogel.  
 National Bureau of Standards, Washington, D.C., R. K. Bell, ASTM-NBS Assistant Research Associate.  
 National-Southwire Aluminum Co., Hawesville, Ky., N. Robinson and E. Gotzy.  
 Ormet Corp., Burnside, La., W. L. Brown and A. D. Lafleur.  
 Reynolds Aluminum Co., Alumina Research Division, Bauxite, Ark., J. B. Ezell, Jr.  
 University of Kentucky, Institute for Mining and Minerals Research, Center for Energy Research Laboratory, Lexington, Ky., T. V. Rebagay.

The overall coordination of the technical measurements leading to certification were performed under the direction of J. I. Shultz, Research Associate, ASTM-NBS Research Associate Program.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. E. Michaelis and R. Alvarez.

## NBS Standard Reference Materials

## BAUXITE SERIES

September 4, 1979

R. E. Michaelis and R. Alvarez, NBS Office of Standard Reference Materials  
and  
J. I. Shultz, ASTM Research Associate

The following table gives the values for four bauxite SRM's that are available in the form of fine powder (<0.08 mm) for use in chemical and instrumental methods of analysis. They are being issued as a culmination of a major Industry-ASTM-NBS cooperative program.

SRM No. Designation	69b Arkansas	696 Surinam	697 Dominican	698 Jamaican
Constituent	Percent by Weight			
Al <sub>2</sub> O <sub>3</sub>	48.8	54.5	45.8	48.2
Fe <sub>2</sub> O <sub>3</sub>	7.14	8.70	20.0	19.6
SiO <sub>2</sub>	13.43	3.79	6.81	0.69
TiO <sub>2</sub>	1.90	2.64	2.52	2.38
ZrO <sub>2</sub>	0.29	0.14	0.065	0.061
P <sub>2</sub> O <sub>5</sub>	0.118	0.050	0.97	0.37
V <sub>2</sub> O <sub>5</sub>	0.028	0.072	0.063	0.064
Cr <sub>2</sub> O <sub>3</sub>	0.011	0.047	0.100	0.080
CaO	0.13	0.018	0.71	0.62
MgO	0.085	0.012	0.18	0.058
MnO	0.110	0.004	0.41	0.38
ZnO	0.0035	0.0014	0.037	0.029
BaO	(0.008) <sup>a</sup>	(0.004)	(0.015)	(0.008)
Na <sub>2</sub> O	(0.025)	(0.007)	(0.036)	(0.015)
K <sub>2</sub> O	0.068	0.009	0.062	0.010
SO <sub>3</sub>	0.63	0.21	0.13	0.22
Loss on Ign.	27.2	29.9	22.1	27.3
Ce	(0.024)	(0.0041)	(0.069)	(0.030)
Co	(0.0001)	(0.00009)	(0.0013)	(0.0045)
Hf	(0.0063)	(0.0032)	(0.0014)	(0.0015)
Sc	(0.0008)	(0.0008)	(0.0058)	(0.0051)
Total	(100.0)	(100.1)	(100.1)	(100.1)

<sup>a</sup>Values in parenthesis are not certified.

The value listed for a certified constituent is the *present best estimate* of the "true" value based on the results of the analytical program for certification (10-12 laboratories). The individual certificates of analysis list the "estimated uncertainties" associated with the certified values (also listed is a semiquantitative mineralogical composition (±5%) as determined by x-ray diffraction studies at the U.S. Geological Survey).

Inquiries regarding the Bauxite SRM's 69b, 696, 697, and 698, should be directed to the Office of Standard Reference Materials, Chemistry Building, B311, National Bureau of Standards, Washington, D.C. 20234. (301) 921-2045.

George A. Uriano, Chief  
Office of Standard Reference Materials

# Certificate of Analysis

## Standard Reference Material 70a

### Feldspar

(All Analyses are Based on Samples Dried 2 hours at 105 °C)

	<i>Percent</i>
Silica (SiO <sub>2</sub> ) .....	67.1
Alumina (Al <sub>2</sub> O <sub>3</sub> ) .....	17.9
Iron (as Fe <sub>2</sub> O <sub>3</sub> ) .....	0.07 <sub>5</sub>
Titania (TiO <sub>2</sub> ) .....	.01
Calcium (as CaO) .....	.11
Barium (as BaO) .....	.02
Sodium (as Na <sub>2</sub> O) .....	2.5 <sub>5</sub>
Potassium (as K <sub>2</sub> O) .....	11.8
Rubidium (as Rb <sub>2</sub> O) .....	0.06
Loss on Ignition .....	.40

Washington, D.C. 20234  
 August 10, 1981  
 (Revision of Certificate dated  
 3-26-65)

George A. Uriano, Chief  
 Office of Standard Reference Materials

U.S. Department of Commerce  
Jumilla M. Kreps  
Secretary

National Bureau of Standards  
Ernest Ambler, Acting Director

# National Bureau of Standards

## Certificate of Analysis

### Standard Reference Material 76a, 77a, and 78a Burnt Refractories

(In Cooperation With the American Society for Testing & Materials)

These materials are in the form of fine powder ( $<0.15$  mm) and are intended for use in checking chemical methods of analysis and in calibration with instrumental methods of analysis.

SRM No. Constituent	76a	77a	78a
	Percent by Weight <sup>a</sup>		
SiO <sub>2</sub>	54.9	35.0	19.4
Al <sub>2</sub> O <sub>3</sub>	38.7	60.2	71.7
Fe <sub>2</sub> O <sub>3</sub>	1.6 <sub>0</sub>	1.0 <sub>0</sub>	1.2
TiO <sub>2</sub>	2.0 <sub>1</sub>	2.6 <sub>0</sub>	3.2 <sub>2</sub>
ZrO <sub>2</sub>	0.15 <sup>c</sup>	0.21	0.31
MgO	.52	.38	.70
CaO	.22	.05	.11
K <sub>2</sub> O	1.33	.09 <sub>0</sub>	1.22
Na <sub>2</sub> O	0.07	.037	.078
P <sub>2</sub> O <sub>5</sub>	.12 <sub>0</sub>	.092	1.3
Li <sub>2</sub> O	.042	.02 <sub>5</sub>	0.12
SrO	.037	.009	.25
Loss on ignition	(.34) <sup>b</sup>	(.22)	(.42)
Total	(100.0)	(100.0)	(100.0)

<sup>a</sup> Based on samples dried at 105 °C for one hour.

<sup>b</sup> A figure in parenthesis is not certified but is given for additional information on the composition.

**CERTIFICATION:** The value listed for a certified constituent is the *present best estimate* of the "true" value based on the results of the analytical program. The value is not expected to deviate from the "true" value by more than  $\pm 1$  in the last significant figure reported. For a subscript figure, the deviation is not expected to be more than  $\pm 5$ . Based on the results of the homogeneity testing, maximum variations within and among samples are estimated to be less than the uncertainty figures given above.

Washington, D.C. 20234

April 5, 1977

(Revision of certificate dated 6-23-76.)

Only change: SrO values from uncertified to certified.)

J. Paul Cali, Chief  
Office of Standard Reference Materials

(Over)

PLANNING, PREPARATION, TESTING, AND ANALYSIS: These replacements for the original Burnt Refractory SRM's were especially prepared and provided to NBS by Harbison-Walker Refractories Company, Garber Research Center, Pittsburgh, Pa., through the efforts of Dr. R. K. Scott.

Carefully selected raw materials were batched to form extruded dobies. The dobies were air dried, oven dried at 120 °C, and then fired at 1427 °C for ten hours in kilns. The fired dobies were crushed and ground and converted to the fine powder product by air classification (about 95 %-325 mesh). The final products were mixed in a blender. At NBS, each of the materials was rebled and resieved.

Homogeneity testing by chemical analyses was performed at NBS by K. M. Sappenfield on selected samples representative of each lot of material for the key constituents, SiO<sub>2</sub> and Fe<sub>2</sub>O<sub>3</sub>.

Standard Deviation of a Single Determination (in wt. %), n = 7

<u>Constituent</u>	<u>.76a</u>	<u>.77a</u>	<u>.78a</u>
SiO <sub>2</sub>	0.09	0.06	0.08
Fe <sub>2</sub> O <sub>3</sub>	.03 <sub>7</sub>	.03 <sub>4</sub>	.03 <sub>8</sub>

Cooperative analyses for certification were performed in the analytical laboratories at Harbison-Walker Refractories Company, Pittsburgh, Pa., R. K. Scott and J. Ryan; and at Pennsylvania State University, College of Earth and Mineral Sciences, University Park, Pa., N. H. Suhr, J. C. Devine, and J. B. Bodkin.

Analyses were performed in the NBS Analytical Chemistry Division by R. K. Bell, O. Menis, T. C. Rains, T. A. Rush, K. M. Sappenfield, M. A. Waguespack, and S. A. Wicks.

The overall responsibility for the technical measurements at NBS was under the direction of W. R. Shields, I. L. Barnes, and O. Menis. The final coordination of the technical measurements was under the direction of J. I. Shultz, Research Associate, ASTM-NBS Research Associate Program.

The technical and support aspects involved in the preparation, certification, and issuance of these SRM's were coordinated through the Office of Standard Reference Materials by R. E. Michaelis.

# National Bureau of Standards

## Certificate of Analysis

### Standard Reference Material 79a

#### Fluorspar

This Standard Reference Material is an acid-grade fluorspar concentrate, to be used primarily for the assay of imported fluorspar for industrial applications. The assay value certified is dependent on the U.S. Customs Laboratory Method given in this Certificate; however, two options exist for the determination of soluble fluoride calculated as calcium fluoride in the acetic acid leach solution. Either the spectrophotometric procedure (Part B), which was used for the original certification of this material, or the ion electrode procedure (Part C), which is included on this Certificate, may be used. The results of the two procedures show excellent agreement.

<u>Constituent</u>	<u>Percent by weight</u>
CaF <sub>2</sub>	97.39 <sup>a</sup> ± 0.06 <sup>b</sup>

<sup>a</sup> Mean value based on 32 determinations using the U.S. Customs Laboratory Method of analysis (attached). The determinations were made by two analysts at each of three Customs laboratories and one NBS laboratory.

<sup>b</sup> Standard deviation of a single determination.

The following values, obtained by quantitative spectrochemical analysis, *are given for information only and are not certified*: Fe, 0.05-0.1%; Al and Sr, 0.01-0.1%; Mg, 0.01-0.05%; Na, 0.001-0.01%; Ba, 0.001-0.005%; K, <0.005%; and Cu, Li, Mn, Pb, and Ti, <0.001%.

The chemically determined value for SiO<sub>2</sub> is 0.67%.

The analytical work leading to certification was performed in the Division of Technical Services, U.S. Customs Laboratories, and by J. R. Moody and K. M. Sappenfield of the NBS Center for Analytical Chemistry. The spectrochemical determinations were made by M. Darr of the NBS Center for Analytical Chemistry.

A comparison of the ion electrode procedure with the spectrophotometric procedure was performed by R. L. Zimmerman, Jr., and H. G. Bertrand of the U.S. Customs Laboratory, New Orleans, La., and by J. R. Moody of the NBS Center for Analytical Chemistry.

This material was supplied by the American Smelting and Refining Company of El Paso, Texas; it was ground to pass a 177 μm (80 mesh) screen.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by J. L. Hague, R. E. Michaelis, and C. L. Stanley.

Washington, D.C. 20234  
January 8, 1980  
(Revision of Certificate  
dated 12-6-71)

George A. Uriano, Chief  
Office of Standard Reference Materials

(over)

#### Method for the Determination of $\text{CaF}_2$ in Fluorspar

**A. Calcium Fluoride Determination:** Transfer a  $0.5 \pm 0.01$  g sample, previously dried at  $100$  to  $105^\circ\text{C}$ , to a 60 mL Pt dish. Add 15 mL acetic acid (1:9) and approximately 50 mg of dry, ashless filter pulp. Digest on a steam bath for 30 minutes and stir with a small glass rod at 5-minute intervals. Stir and filter through an 11-cm close-texture filter paper (S&S No. 589 Red Ribbon, or equal) to which has been added approximately 4 mL filter pulp slurry (1 g ash-free pulp in 100 mL of water). Thoroughly wash crucible, stir rod, and paper with small portions of hot water (approximately 35 mL). The filtrate and washings should be collected in a polyethylene bottle for the determination of dissolved fluoride by either the spectrophotometric method described in Part B or the ion-selective electrode method described in Part C. Transfer paper and residue to original crucible, wipe the funnel with a small piece of filter paper, and add the paper to the crucible to assure recovery of fine particles. Dry crucible and contents, ignite to a dull red heat ( $600^\circ\text{C}$ ), and cool to room temperature.

Transfer the residue, as completely as possible, from the crucible to a 400-mL beaker by gently tapping the crucible. Add 5 to 10 mL HCl to the crucible and warm on a steam bath or a temperature-regulated sand bath. Transfer the HCl to the beaker containing the residue. Police and wash the crucible with a minimum amount of hot water. Repeat adding HCl and washing 2 or 3 times using a total of 25 mL HCl. Add approximately 0.5 g of crystalline boric acid to beaker, cover with a watch glass, and digest on the steam bath for 15 minutes. Remove the beaker and cool to room temperature. Add 0.5 mL  $\text{HNO}_3$  and slowly evaporate on the steam bath to approximately 10 mL. During evaporation, the cover glass should be gradually removed from the beaker. Wash down the sides of the beaker with a fine stream of water and adjust the volume to approximately 75 mL. Heat to  $70$  to  $80^\circ\text{C}$ , remove from heat, and add 100 mL of precipitating solution [3.8 g ammonium oxalate, 1.1 g oxalic acid, and 0.05 g disodium ethylenediaminetetraacetate (EDTA) in 100 mL of water]. If a white precipitate forms at this point, add HCl dropwise until it dissolves.

Heat to boiling and slowly add  $\text{NH}_4\text{OH}$  (1:1) until a heavy white precipitate forms. Add 1 mL of bromocresol green solution (0.1 g soluble salt in 100 mL of water) and continue the addition until the color of the solution changes from yellowish-orange to grayish-green. Digest for 30 minutes on the steam bath, let stand for 60 minutes at room temperature, and filter by decantation through a prepared Gooch crucible (size 3, prepared with 2.1 cm glass-fiber filter paper, H. Reeve Angel No. X-934AH, or equivalent). Wash 3 or 4 times with a cold wash solution (0.2 g ammonium oxalate and 0.1 g oxalic acid in 100 mL of water) and finally with 3 or 4 10-mL portions of cold water. (The washed precipitate should be free of chloride, ammonium oxalate, or any other contaminant that might reduce the  $\text{KMnO}_4$  solution.) Transfer the contents of the crucible to a 400-mL beaker, add 250 mL  $\text{H}_2\text{SO}_4$  (1:19), and adjust the solution temperature to  $27 \pm 3^\circ\text{C}$ . While stirring, add approximately 90 percent of the 0.15 N  $\text{KMnO}_4$  solution to be used for titration. This should be added at a rate of approximately 30 mL per minute from a buret. Allow to stand until the pink color disappears, e.g., approximately 45 seconds. (If pink color persists, too much  $\text{KMnO}_4$  solution has been added and the determination must be repeated.) Heat to  $57 \pm 3^\circ\text{C}$  and complete the titration by slowly adding the last 0.5 to 1 mL  $\text{KMnO}_4$  solution dropwise and allowing each drop to decolorize completely before adding the next drop. The end point is a pink color persisting for 30 seconds or more. See calculations.

**B. Determination of Fluoride in Acetic Acid Leach Solution:** Remove all ions from the solution that might cause interference in the spectrophotometric determination of dissolved fluoride, by using either the ion-exchange method, or the fluorine-distillation method described in 2 and 3. Prepare a reagent blank, by substituting 15 mL of acetic acid (1:9), diluted to approximately 55 mL with distilled water and analyze simultaneously with sample.

**1. Preparation of calibration curve:** Prepare a dilute fluoride standard ( $1 \text{ mL} \approx 0.1 \text{ mg CaF}_2$ ) from a standard solution ( $1 \text{ mL} \approx 1 \text{ mg CaF}_2$ ) of 1.0755 g NaF diluted to one liter in a polypropylene volumetric flask with distilled water. Take a series of aliquots of 1, 2, 4, 6, 8, 11, 16 mL, etc., from the diluted fluoride standard and transfer them to 60-mL polyethylene bottles. Add 15 mL of acetic acid (1:9) and sufficient distilled water to bring the final volume to approximately 55 mL. Proceed to the ion-exchange or fluorine-distillation method.



2. Ion-exchange method: Prepare an anion-exchange column by pouring a slurry of distilled water and Rexyn 201 (OH) resin, or equivalent, sufficient to give a total exchange capacity of 27 to 30 meq. into a 25 mL polystyrene buret plugged with washed absorbent cotton (not glass wool) and drain off the excess water without allowing the water level to fall below the top of the resin column at any time. Continue this procedure until the resin is within about 5 mL of the top of the buret. Wash the resin with 20 mL N NaOH, and finally with distilled water (approximately 100 mL), until the effluent is no longer alkaline. Adjust the stopcock to give a flow rate of approximately 100 drops per minute. The flow rate is an indicator of the state of the column. Backwash with distilled water whenever the flow rate falls below 100 drops per minute. Resin must be covered with liquid at all times.

Prepare a cation-exchange column in the same manner as the anion exchange column using a slurry of distilled water and Rexyn 101(H) resin, or equivalent, sufficient to give a total exchange capacity of 40 to 45 meq. Wash the resin with 50 mL of 1N HCl, and finally with distilled water (approximately 200 mL) until the effluent is free of chloride ion. Flow rate should be approximately 100 drops per minute.

Each of the diluted aliquots, as well as the blank, is run separately as follows: After completing both columns, pass the entire volume from the polyethylene bottle through the anion-exchange column, always maintaining the liquid level slightly above the top of resin. Wash the polyethylene bottle with distilled water and pass washings through the column using a total of approximately 60 mL for each sample. Discard the effluent and washings. Recover the fluoride, acetate, and other anions from the column by passing 20 mL of 1N NaOH through the column at a flow rate of approximately 100 drops per minute. Collect the effluent in the original polyethylene bottle. Wash the column with distilled water and collect washings in the same bottle to capacity (60 mL). Pour the contents of this bottle, and water used to rinse it, into the cation column and collect the effluent in a 100-mL volumetric flask. Wash the column with distilled water and collect just short of the mark in the same flask. Bring the liquid to the mark with distilled water. Proceed to the spectrophotometric determination.

3. Distillation method: Have water in a steam generator actively boiling, but do not connect to the fluoride distillation apparatus (Willard and Winter, or equivalent) at this time. Distill each of the diluted aliquots as well as the blank, separately as follows: Transfer the entire volume from the polyethylene bottle to the 500-mL Claisen flask of the distillation apparatus using a minimum quantity of water for rinsing. Add 50 mL  $\text{H}_2\text{SO}_4$  (1:1) and a few glass beads. Insert a stopper carrying the steam tube and thermometer, and heat the flask with a Bunsen burner or an electric heater. Connect the condenser at once and place a 200-mL polypropylene volumetric flask at the receiving end. Water soon begins to distill over and the temperature of the liquid rises as the  $\text{H}_2\text{SO}_4$  concentration increases. When the temperature reaches  $120^\circ\text{C}$  connect the flask to the steam generator by means of the rubber tube. The rate of steam generation and the rate at which the Claisen flask is being heated should be regulated so that the temperature of the liquid in the distilling flask is maintained at  $135$  to  $145^\circ\text{C}$ . Distill until the total volume in the receiver is approximately 185 mL (Note 1). Disconnect the condenser and rinse with a small portion of water, catching the washings in the flask containing the distillate, and fill to mark with distilled water. Proceed to the spectrophotometric determination.

4. Spectrophotometric determination: Pipette about one-tenth of the volume of the solution from steps B-2 or B-3 containing approximately 0.1 to 0.2 mg  $\text{CaF}_2$  (not more than 50 mL) into a 100-mL polypropylene volumetric flask and add 15 mL 0.1 N sodium acetate solution and 25 mL methyl cellosolve. Bring the solution level in the flask almost to mark with distilled water and allow to cool to room temperature (mixing reaction is slightly exothermic) before making a final volume adjustment. (Resulting solution should have a pH of 3 to 5; optimum color development occurs in this range.)

Pour the entire contents of the volumetric flask into a 125-mL stoppered erlenmeyer flask containing 0.1 g of thorium chloranilate (Note 2). Stopper the flask and place on a shaking apparatus. Shake for 55 minutes, remove, and allow the contents of the flask to settle for 5 minutes. Filter through a close-texture filter paper, discarding the first 5 to 10 mL of filtrate.

Read the absorbance of sample against the reagent blank using 1 cm absorption cells in a Beckman DU spectrophotometer, or its equivalent, at a wavelength of 330 nm and a slit width of about 0.2 mm. Plot absorbance vs mg of  $\text{CaF}_2$ . This should yield a straight line passing through the origin.



5. Analysis of acetic acid leach solution: Carry the entire filtrate from the acetic acid digestion through steps B-2 or B-3 and finally B-4. Determine mg of  $\text{CaF}_2$  by using the calibration curve and proceed with calculation.

C. Determination of Fluoride in Acetic Acid Leach Solution with an Ion-Selective Electrode.

1. Apparatus, Reagents, and Solutions

a. Special Apparatus

- 1) Fluoride specific ion electrode
- 2) Single junction reference electrode
- 3) Expanded scale pH meter with millivolt capability

b. Reagents

ACS Reagent grades of sodium fluoride, glacial acetic acid, sodium chloride, and sodium hydroxide, must be used. For (1,2 Cyclohexylenedinitrilo)- tetraacetic acid (CDTA), a practical grade available from a commercial source may be used.

c. Solutions

1) TISAB II - (Total Ionic Strength Adjustment Buffer)

To 500 mL of distilled water in an 800 mL beaker, add 57 mL of glacial acetic acid, 58 g of sodium chloride, and 4 g of CDTA. Place the beaker in a cold water bath, on a magnetic stirrer. Stir the mixture, while adding concentrated sodium hydroxide, to provide a pH between 5.0 and 5.5. Transfer the contents of the beaker to a 1-liter flask, and dilute to the mark with distilled water. (TISAB II is used to provide a constant ionic strength background, decomplex the fluoride ions, and adjust and buffer the pH of the solution.)

2) Standards

- a) Stock Solution ( $1900 \text{ ppmF}^-$ ) - Dissolve 4.199 g of sodium fluoride in a 1-liter polypropylene volumetric flask and dilute to volume with distilled water.
- b) Working stock solution ( $19.00 \text{ ppmF}^-$ ) - Transfer 10 mL of stock solution (a) to a 1-liter polypropylene volumetric flask and dilute to the mark with distilled water.
- c) Working standards - Prepare standards according to Table I.

TABLE I

$\text{F}^-$ Conc. ( $\mu\text{g/mL}$ )	Working Stock (mL)	TISAB II (mL)	Distilled Water <sup>1</sup> (mL)
9.50	50.0	50	- -
4.75	25.0	50	25
2.85	15.0	50	35
1.90	10.0	50	40
0.95	5.00	50	45
0.38	2.00	50	48

<sup>1</sup>If prepared in a 100 mL flask, dilute to mark with distilled water.

Store working standards in polyethylene bottles and refrigerate until use.

2. Procedure

Transfer the contents of the polyethylene bottles containing the acetic acid wash (Section A) to 200 mL polyethylene volumetric flasks and dilute to volume with distilled water. Transfer a 25 mL aliquot to a 50 mL polyethylene volumetric flask and dilute to volume with TISAB II. Place in refrigerator until ready for use. In a polyethylene bottle, prepare a reagent blank consisting of 15 mL of acetic acid (1:9) and approximately 55 mL of distilled water.

Transfer the working standards, test solutions, and the reagent blank to 150 mL polyethylene beakers and allow to come to room temperature. Stir all solutions with magnetic stirrers. Adjust the pH meter to -150 mV by use of the 19.00 ppm  $F^-$  working stock solution. Record the values of the solutions beginning with the least concentrated working standard. NOTE: For solutions containing low concentrations of fluoride, the pH meter may require approximately two minutes to stabilize (cessation of drift). Rinse and dry the electrodes with tissue paper between readings. Record the values of the test solutions and the reagent blank immediately following the working standards.

Plot a curve from the values obtained for the working standards, and determine the concentration of the test solutions from the curve. Proceed with calculations.

#### D. Calculations:

First, calculate the %  $CaF_2$  found in Part A. Then calculate the %  $CaF_2$  found in either Part B (spectrophotometric procedure) or Part C (electrode procedure). Add the value calculated for either Part B or C to that calculated for Part A, to determine the total %  $CaF_2$  in the sample.

$$\text{For Part A: } \% CaF_2 = \frac{3.904 V_1 N}{W_s}$$

where:  $V_1$  = Volume of  $KMnO_4$  solution used (corrected for blank)

$N$  = Normality of  $KMnO_4$  solution

$W_s$  = gram weight of sample

$$\text{For Part B: } \% CaF_2 = \frac{0.1 W_c F}{W_s}$$

where:  $W_c$  = milligram weight of  $CaF_2$  in aliquot

$F$  = aliquot factor;  $\frac{\text{Total volume from B-2 or B-3}}{\text{volume used}}$

$W_s$  = gram weight of sample

$$\text{For Part C: } \% CaF_2 = \frac{CV_2AG}{W_s} \times 10^{-4}$$

where:  $C$  = observed concentration in ppm  $F^-$  ( $\mu g F^-/mL$ )

$V_2$  = volume of test solution

$A$  = aliquot factor;  $\frac{\text{total volume}}{\text{volume used}}$

$G$  = gravimetric factor;  $\frac{CaF_2}{2F}$

$W_s$  = gram weight of sample

For the conditions described in Part C, this formula may be simplified as follows:

1) The sample is diluted to 200 mL and a 25 mL aliquot is taken; thus the aliquot factor, A, is:  $A = \frac{200}{25} = 8.00$

2) The volume of test solution  $V_2$  is always 50 mL;  $V_2 = 50.0$  mL

3) The gravimetric factor, G, is:  $G = \frac{\text{CaF}_2}{2\text{F}} = \frac{78.0768}{37.9968} = 2.055$

by substitution:

$$\% \text{CaF}_2 = \frac{C \times 50.0 \times 8.00 \times 2.055}{W_s} \times 10^{-4} \text{ or}$$

$$\% \text{CaF}_2 = \frac{0.0822C}{W_s}$$

Note 1. The volume of distillate necessary for complete recovery of fluoride will vary with the capacity of the distillation apparatus. Calibrate the apparatus by distilling a known quantity of fluoride to various final volumes to determine the volume necessary for complete recovery.

Note 2. Chloranilates are supplied as dry powders of high stability. However, long storage may reduce activity. Drying at 105 °C for at least 4 hours restores activity. Keep bottles tightly capped to prevent moisture absorption, which reduces activity and hinders the color development.

U.S. Department of Commerce  
Juanita M. Kreps  
Secretary  
National Bureau of Standards  
Ernest Ambler, Acting Director

# National Bureau of Standards

## Certificate of Analysis

### Standard Reference Material 81a

#### Glass Sand

(In Cooperation with the American Society for Testing and Materials)

This SRM is issued in the form of a ground powder (95% less than 106  $\mu\text{m}$ ) blended to ensure homogeneity. It should be dried for 2 hours at 105 °C before use.

<u>Constituent</u>	<u>Recommended Value</u>		<u>s</u>
	<u>Percent by Weight</u>	<u>Range</u>	
Al <sub>2</sub> O <sub>3</sub>	0.66	0.62 - 0.69	0.011
Fe <sub>2</sub> O <sub>3</sub>	.082	.075 - .089	.0024
TiO <sub>2</sub>	.12	.10 - .14	.0064
ZrO <sub>2</sub>	.034	.025 - .042	.0026
Cr <sub>2</sub> O <sub>3</sub>	46 $\mu\text{g/g}$	33 - 58	3.9

Certification - The recommended value listed for each oxide is the best estimate of the true value based on the analytical data from both cooperators and NBS. The range of values listed is the tolerance interval, constructed such that it will cover at least 95% of the population with a probability of 0.99. It is computed as  $\bar{X} \pm Ks$ ; where  $s$  is the standard deviation,  $K$  is a factor that depends on  $n$  (the number of samples measured),  $p$ , the proportion of the total sample covered (95%), and  $\gamma$ , the probability level (99%). In all cases none of the  $n$  values used exceeded the range specified. Thus, it includes variability between laboratories and between samples.

The overall direction and coordination of the round-robin analysis leading to certification were performed by Paul Close, Chairman of ASTM Subcommittee C-14.02 on Chemical Analysis of Glass and Glass Products.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by W. P. Reed.

Washington, D.C. 20234  
January, 1978

J. Paul Cali, Chief  
Office of Standard Reference Materials

(over)

Chemical analyses for certification were performed in the following laboratories:

Anchor Hocking Corp., Lancaster, Ohio, R. E. Carr  
Brockway Glass Co., Inc., Brockway, Pa., E. L. McKinley.  
Corning Glass Works, Corning, N.Y., Y. S. Su.  
Ford Motor Co., Lincoln Park, Mich., T. O. LaFramboise.  
National Bureau of Standards, Analytical Chemistry Division, E. J. Maienthal, J. D. Messman and T. C.  
Rains.  
Kimble Div. Owens-Illinois, Vineland, N. J., H. S. Moser.  
Owens-Illinois, Inc., Toledo, Ohio, P. Close.  
Penn State Univ., University Park, Pa., J. B. Bodkin.

# Certificate of Analysis

## Standard Reference Material 88a

### Dolomitic Limestone

(All analyses are based on samples dried two hours at 110 °C)

	<i>Percent</i>
Silica (SiO <sub>2</sub> ) -----	1.20
Alumina (Al <sub>2</sub> O <sub>3</sub> ) -----	0.19
Total Iron (as Fe <sub>2</sub> O <sub>3</sub> ) -----	.28
Titania (TiO <sub>2</sub> ) -----	.02
Manganese (as MnO) -----	.03
Calcium (as CaO) -----	30.1 s
Strontium (as SrO) -----	0.01
Magnesium (as MgO) -----	21.3
Sodium (as Na <sub>2</sub> O) -----	0.01
Potassium (as K <sub>2</sub> O) -----	.12
Phosphorus (as P <sub>2</sub> O <sub>5</sub> ) -----	.01
Carbonate (as CO <sub>2</sub> ) -----	46.6
Loss on ignition -----	46.7

Washington, D.C. 20234  
 July 22, 1982  
 (Revision of Certificate  
 dated 1-31-67)

George A. Uriano, Chief  
 Office of Standard Reference Materials

# Certificate of Analysis

## STANDARD REFERENCE MATERIAL 97 a

### Flint Clay

(Results based on sample dried for two hours at 140 °C)

Analyst	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	K <sub>2</sub> O	Na <sub>2</sub> O	Li <sub>2</sub> O	ZrO <sub>2</sub>	BaO	MgO	CaO	SrO	Cr <sub>2</sub> O <sub>3</sub>	Loss on Ignition
1 <sup>[1]</sup> .....	43.74	38.65	{0.45 <sup>a</sup> .46 <sup>b</sup> }	{1.88 <sup>c</sup> 1.89 <sup>d</sup> }	0.34	0.53 <sup>e</sup>	0.033 <sup>f</sup>	0.12 <sup>g</sup>	0.063 <sup>h</sup>	0.078 <sup>i</sup>	0.16 <sup>j</sup>	0.11 <sup>k</sup>	0.17 <sup>l</sup>	0.028 <sup>m</sup>	13.32
2 <sup>[1]</sup> .....	43.68	38.95	.45	1.95	.35	.51 <sup>e</sup>	.041 <sup>f</sup>	.10 <sup>g</sup>	.....	.07	.14 <sup>j</sup>	.11 <sup>k</sup>	.18 <sup>l</sup>	.03	13.31
3.....	43.60	38.79	.43 <sup>a</sup>	1.87 <sup>d</sup>	.38 <sup>e</sup>	.46 <sup>f</sup>	.....	.....	.....	.....	.....	.....	.....	.....	.....
Average...	43.67	38.79	0.45	1.90	0.36	0.50	0.037	0.11	.....	0.07 <sub>5</sub>	0.15	0.11	0.18	0.03	13.32

References: [1] G.E.F. Lundell and J.I. Hoffman, NBS J. Res. 1, 91 (1928) RP5.  
[2] L. C. Peck, Geological Survey Bulletin 1170, (1964).

<sup>a</sup>o-phenanthroline photometric method.  
<sup>b</sup>Iron reduced with SnCl<sub>2</sub> and titrated with standard potassium dichromate solution.  
<sup>c</sup>Cupferon gravimetric method.  
<sup>d</sup>H<sub>2</sub>O<sub>2</sub> photometric method.

<sup>e</sup>Flame emission spectrometric method.  
<sup>f</sup>Pyrocatechol violet photometric method.  
<sup>g</sup>Atomic absorption method.  
<sup>h</sup>Diphenylcarbazide photometric method.  
<sup>i</sup>Molybdenum-blue photometric method.

### List of Analysts

1. R. K. Bell, B. B. Bendigo, T. C. Rains, T. A. Rush, E. R. Deardorff, J. R. Baldwin, R. A. Paulson, W. P. Schmidt, and S. D. Rasberry, Analytical Chemistry Division, Institute for Materials Research, National Bureau of Standards.
2. L. C. Peck, United States Geological Survey, Denver, Colorado.
3. L. M. Melnick, J. D. Selvaggio, and D. G. Cunningham, Applied Research Laboratory, United States Steel Corporation, Pittsburgh, Pennsylvania.

The material for the preparation of this standard was provided by the A. P. Green Fire Brick Company, Mexico, Missouri.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanships of O. Menis and J. I. Shultz.

The technical and support aspects involved in the preparation, certification and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by J. L. Hague.

Washington, D. C. 20234  
October 8, 1969

J. Paul Cali, Acting Chief  
Office of Standard Reference Materials

U. S. Department of Commerce  
Maurice H. Stans  
Secretary

National Bureau of Standards  
L. M. Branscomb, Director

# Certificate of Analysis

## STANDARD REFERENCE MATERIAL 98a

### Plastic Clay

(Results based on sample dried for two hours at 140 °C)

Analyst	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	K <sub>2</sub> O	Na <sub>2</sub> O	Li <sub>2</sub> O	ZrO <sub>2</sub>	BaO	MgO	CaO	SrO	Cr <sub>2</sub> O <sub>3</sub>	Loss on Ignition
1 <sup>(1)</sup> .....	48.98	33.13	$\frac{1.34^1}{1.37^2}$	$\frac{1.56^1}{1.63^2}$	0.11	1.07 <sup>c</sup>	0.080 <sup>c</sup>	0.075 <sup>c</sup>	0.042 <sup>c</sup>	0.031 <sup>c</sup>	0.42 <sup>c</sup>	0.31 <sup>c</sup>	0.041 <sup>c</sup>	0.030 <sup>b</sup>	12.40
2 <sup>(2)</sup> .....	48.91	33.31	1.35	1.64	.10	1.08 <sup>c</sup>	.083 <sup>c</sup>	.064 <sup>c</sup>	.....	.03	.43 <sup>c</sup>	.31 <sup>c</sup>	.037 <sup>c</sup>	.04	12.49
3.....	.....	33.12	1.28 <sup>c</sup>	1.61 <sup>d</sup>	.11 <sup>c</sup>	0.98 <sup>c</sup>	.....	.....	.....	.....	.....	.....	.....	.....	.....
Average..	48.94	33.19	1.34	1.61	0.11	1.04	0.082	0.070	.....	0.03	0.42	0.31	0.039	0.03	12.44

References: [1] G.E.F. Lundell and J.I. Hoffman, NBS J. Res. 1, 91 (1928) RP5.  
[2] L.C. Peck, Geological Survey Bulletin 1170, (1964).

<sup>a</sup>o-phenanthroline photometric method.  
<sup>b</sup>Iron reduced with SnCl<sub>2</sub> and titrated with standard potassium dichromate solution.  
<sup>c</sup>Cupferron gravimetric method.  
<sup>d</sup>H<sub>2</sub>O<sub>2</sub> photometric method.

<sup>e</sup>Flame emission spectrometric method.  
<sup>f</sup>Pyrocatechol violet photometric method.  
<sup>g</sup>Atomic absorption method.  
<sup>h</sup>Diphenylcarbazide photometric method.  
<sup>i</sup>Molybdenum-blue photometric method.

#### List of Analysts

1. R. K. Bell, B. B. Bendigo, T. C. Rains, T. A. Rush, E. R. Deardorff, J. R. Baldwin, R. A. Paulson, W. P. Schmidt, and S. D. Rasberry, Analytical Chemistry Division, Institute for Materials Research, National Bureau of Standards.
2. L. C. Peck, United States Geological Survey, Denver, Colorado.
3. L. M. Melnick, J. D. Selvaggio, and D. G. Cunningham, Applied Research Laboratory, United States Steel Corporation, Pittsburgh, Pennsylvania.

The material for the preparation of this standard was provided by the A. P. Green Fire Brick Company, Mexico, Missouri.

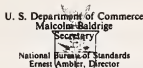
The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of O. Menis and J. I. Shultz.

The technical and support aspects involved in the preparation, certification and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by J. L. Hague.

Washington, D. C. 20234  
October 8, 1969

J. Paul Cali, Acting Chief  
Office of Standard Reference Materials





# Certificate of Analysis

## Standard Reference Material 99a

### Feldspar

(All Analyses are Based on Samples Dried 2 hours at 105 °C)

	Percent
Silica ( $\text{SiO}_2$ ) .....	65.2
Alumina ( $\text{Al}_2\text{O}_3$ ) .....	20.5
Iron (as $\text{Fe}_2\text{O}_3$ ) .....	0.06
Titania ( $\text{TiO}_2$ ) .....	.007
Calcium (as $\text{CaO}$ ) .....	2.14
Barium (as $\text{BaO}$ ) .....	0.26
Magnesium (as $\text{MgO}$ ) .....	.02
Sodium (as $\text{Na}_2\text{O}$ ) .....	6.2
Potassium (as $\text{K}_2\text{O}$ ) .....	5.2
Phosphorus (as $\text{P}_2\text{O}_5$ ) .....	0.02
Loss on Ignition .....	0.26

Washington, D.C. 20234  
August 10, 1981  
(Revision of Certificate dated  
3-26-65)

George A. Uriano, Chief  
Office of Standard Reference Materials

UNITED STATES DEPARTMENT OF COMMERCE  
WASHINGTON 25, D.C.

National Bureau of Standards  
Certificate of Analyses  
of  
Standard Sample No. 103 a  
Chrome Refractory

(All results are based on a sample dried for 2 hours at 105 to 110° C)

Analyst	Cr <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	Total iron as FeO	MnO	MgO	CaO	SiO <sub>2</sub>	TiO <sub>2</sub>	ZrO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>
1.	<sup>a</sup> 32.05	<sup>b</sup> 29.95	<sup>c</sup> 12.43	<sup>d</sup> 0.12	<sup>e</sup> 18.57	<sup>f</sup> 0.70	<sup>g</sup> 4.68	<sup>h</sup> 0.22	<sup>i</sup> 0.01	<sup>j</sup> 0.007
2.	<sup>k</sup> 31.95	<sup>l</sup> 30.10 <sup>m</sup> 30.01	<sup>n</sup> 12.43	<sup>o</sup> .12	<sup>p</sup> 18.50	<sup>q</sup> .69	<sup>r</sup> 4.68	<sup>s</sup> .21		<sup>t</sup> .01
3.	<sup>u</sup> 32.07 <sup>v</sup> 32.09	<sup>w</sup> 30.02 <sup>x</sup> 29.95	<sup>y</sup> 12.45	<sup>z</sup> .11	<sup>aa</sup> 18.51	<sup>ab</sup> { ".68" ".67"	<sup>ac</sup> 4.59	<sup>ad</sup> .22		<sup>ae</sup> <.004
4.	<sup>af</sup> 32.08	<sup>ag</sup> 30.05		<sup>ah</sup> .10	<sup>ai</sup> 18.62	<sup>aj</sup> .60	<sup>ak</sup> 4.58	<sup>al</sup> .20		<sup>am</sup> .017
5.	<sup>an</sup> 31.98		<sup>ao</sup> 12.43				<sup>ap</sup> 4.63			
6.	<sup>aq</sup> 32.10	<sup>ar</sup> 29.91	<sup>as</sup> 12.40	<sup>at</sup> .09	<sup>au</sup> 18.45	<sup>av</sup> .72	<sup>aw</sup> 4.64	<sup>ax</sup> .21	<sup>ay</sup> .01	<sup>az</sup> .01
7.	<sup>ba</sup> 32.12	<sup>bb</sup> 29.85		<sup>bc</sup> .10	<sup>bd</sup> 18.49	<sup>be</sup> .70		<sup>bf</sup> .25		
8.	<sup>bg</sup> 31.78	<sup>bh</sup> 29.80	<sup>bi</sup> 12.43	<sup>bj</sup> .10	<sup>bk</sup> 18.63	<sup>bl</sup> .74	<sup>bm</sup> 4.60	<sup>bn</sup> .23		<sup>bo</sup> { ".009" ".009"
Average.....	32.06	29.96	12.43	0.11	18.54	0.69	4.63	0.22	0.01	0.01

<sup>a</sup> Peroxide oxidation and potentiometric titration with ferrous ammonium sulfate. Corrected for vanadium.  
<sup>b</sup> Dissolved in perchloric acid, volatilized chromium with hydrochloric acid, extracted  $PbO_3$  cupferrate with chlorine at pH 1.5, separated aluminum by extracting  $R_2O_3$  cupferrate in 4N hydrochloric acid with chloroform, precipitated aluminum with ammonia and weighed as  $Al_2O_3$ .  
<sup>c</sup> Dissolved in perchloric acid.  $SnCl_4$ - $K_2Cr_2O_7$ .  
<sup>d</sup> Dissolved in perchloric acid. Peroxide colorimetric method.  
<sup>e</sup> Dissolved in perchloric acid. Gravimetric phosphate method. Corrected for Ca and Mn.  
<sup>f</sup> Precipitated with magnesium, separated as sulfate in alcohol, precipitated with oxalate and weighed as  $CaO$ .  
<sup>g</sup> Perchloric acid dehydration.  
<sup>h</sup> Peroxide colorimetric method. Used 2d chloroform extraction footnote "b".  
<sup>i</sup> Used  $PbO_3$  precipitate, separated with cupferron, and determined gravimetrically as phosphate.  
<sup>j</sup> Dissolved in perchloric acid, precipitated with ammonia and determined by molybdenum blue colorimetric method.  
<sup>k</sup> Average of footnote "m" and leached sodium peroxide fusion with water, filtered, acidified filtrate, added excess ferrous ammonium sulfate and titrated with permanganate.  
<sup>l</sup> Precipitated with phenylhydrazine. Corrected for  $Cr_2O_3$ ,  $Fe_2O_3$ ,  $TiO_2$ , and  $P_2O_5$ .  
<sup>m</sup> Separated with cupferron, corrected for  $Cr_2O_3$  and  $P_2O_5$ .  
<sup>n</sup> Leach sodium peroxide fusion, filtered and dissolved precipitate.  $SnCl_4$ - $K_2MnO_4$ .  
<sup>o</sup> Perchloric acid-sulfuric acid dehydration.  
<sup>p</sup> Reduced with excess ferrous iron and titrated with permanganate.  
<sup>q</sup> Reduced with excess ferrous iron and titrated with dichromate. Corrected for vanadium.  
<sup>r</sup> Separated with ammonia and mercury cathode and precipitated with ammonia. Corrected for  $TiO_2$ .

<sup>s</sup> Removed chromium by ion exchange, separated with ammonia and mercury cathode and precipitated with ammonia. Corrected for  $TiO_2$ .  
<sup>t</sup> Leached sodium peroxide-sodium hydroxide fusion, reprecipitated with ammonia.  $SnCl_4$ - $K_2Cr_2O_7$ .  
<sup>u</sup> Leached sodium peroxide fusion and dissolved hydroxides. Peroxide colorimetric method.  
<sup>v</sup> Used filtrate of ammonia precipitation (footnote "m"), precipitated as phosphate, separated as sulfate in alcohol and determined as oxalate.  
<sup>w</sup> Fused with sodium peroxide and dehydrated with perchloric acid.  
<sup>x</sup> Fused with sodium peroxide. Ammonia and cupferron separations made. Peroxide colorimetric method.  
<sup>y</sup> Fused with sodium peroxide and reduced chromium with alcohol. Phosphomolybdate volumetric method.  
<sup>z</sup> Leached sodium peroxide fusion, precipitated with  $NH_4Cl$ , reprecipitated with ammonia, dehydrated with perchloric acid, filtered, and precipitated with ammonia. Dissolved in perchloric acid and volatilized chromium with hydrochloric acid. Volumetric bismuthate method.  
<sup>aa</sup> Volumetrically. Oxalate titrated with permanganate.  
<sup>ab</sup> Dissolved in perchloric acid and made ammonium separation. Peroxide colorimetric method.  
<sup>ac</sup> Dissolved in perchloric acid, precipitated with ammonium molybdate, reduced with Jones reductor and titrated with permanganate.  
<sup>ad</sup> Oxidized with permanganate and titrated potentiometrically with ferrous ammonium sulfate. Corrected for vanadium.  
<sup>ae</sup>  $SiO_2$  removed,  $P_2O_5$  precipitated with  $NH_4OH$ ,  $Al_2O_3$  cupferron separation, aluminum precipitated in filtrate with cupferron after neutralization with  $NH_4OH$ . Corrected for  $Cr_2O_3$ .  
<sup>af</sup> Dissolved in perchloric and sulfuric acids, precipitated as sulfide, ignited, fused with potassium bisulfate, oxidized with permanganate and titrated with titanous chloride.

<sup>ag</sup> Dissolved in perchloric and sulfuric acids, precipitated with magnesium and calcium, removed calcium with alcoholic sulfate separation. Bismuthate method.  
<sup>ah</sup> Used acid cupferrates of aluminum determination (footnote "f") and made an ammoniacal titration, hydrogen sulfide separation. Peroxide colorimetric method.  
<sup>ai</sup> Gravimetric phosphomolybdate method.  
<sup>aj</sup> Fused with sodium carbonate-sodium borate and made a mercury cathode separation. Gravimetric 8-hydroxyquinoline method. Corrected for titanium.  
<sup>ak</sup> Dissolved in perchloric and sulfuric acids, precipitated with ammonia, and dissolved in hydrochloric acid.  $SnCl_4$ - $K_2Cr_2O_7$ .  
<sup>al</sup> In filtrate from silicon determination reduced chromium with sulfurous acid, reoxidized iron with nitric acid, precipitated with ammonia, made sodium carbonate separation and determined photometrically with periodate.  
<sup>am</sup> Gravimetric as phosphate. Used filtrate from CaO determination.  
<sup>an</sup> Prepared solution as in footnote "m". Peroxide colorimetric method.  
<sup>ao</sup> Dissolved in nitric, hydrochloric and perchloric acids, filtered, volatilized arsenic as bromide, and precipitated phosphorus as phosphomolybdate. Determined the equivalent molybdenum as lead molybdate.  
<sup>ap</sup> Dissolved in perchloric acid, volatilized arsenic as bromide and determine phosphorus colorimetrically as phospho-vanado-molybdate in iso-amyl alcohol.  
<sup>aq</sup> United from average.  
<sup>ar</sup> Analyst 1 and analyst 3 reported 0.09 percent vanadium and analyst 6 reported 0.12 percent  $V_2O_5$ .  
<sup>as</sup> Combination in a lake furnace at approximately 900° C indicates 0.97 percent of water which is not removed by drying at 105 to 110° C.

List of Analysts

- Keith M. Sappenfield, National Bureau of Standards.
- Paul J. Byler, Booth, Garrett & Blair, Philadelphia, Pa.
- Andrew S. McCreath & Sons, Inc., Harrisburg, Pa.
- John H. Montague, E. G. Lavino and Co., Norristown, Pa.
- George Oplinger, K. A. Lane, and M. S. Budd, Solvay Process Division, Allied Chemical Corp., Syracuse, N.Y.
- B. C. Ruprecht and R. P. Lucas, Harbison-Walker Refractories Co., Pittsburgh, Pa.
- L. J. Trostel, General Refractories Co., Baltimore, Md.
- C. E. A. Shanahan, Richard Thomas & Baldwins, Ltd., Whitchurch, Aylesbury, Bucks, England.

WASHINGTON, D.C., September 28, 1962.

V. A. ASTIN, Director.

GPO 565340

# National Bureau of Standards Certificate of Analysis Standard Reference Materials 113a and 329 Zinc Concentrates

These Standard Reference Materials are in the form of fine powder (<15 mm) and are intended for use both in checking chemical methods of analysis and in calibration with instrumental methods of analysis.

**CAUTION:** The bottle should be kept tightly closed except when in direct use. Store in a desiccator over desiccant.

SRM No. Element	113a Percent by Weight <sup>a</sup>	329 Percent by Weight <sup>a</sup>
Zinc	57.3	45.5
Lead	2.80	6.06
Iron	2.08	12.94
Calcium Oxide	1.19	0.08
Magnesium Oxide	0.75	.165
Cadmium	.78	.14
Copper	.31	.132
Cobalt	(.11) <sup>b</sup>	(.009)
Nickel	(.07)	(.006)
Sulfur (Total)	30.6	(31.7)
Silicon Dioxide	(1.54)	(0.61)
Indium	... <sup>c</sup>	.019
Silver	0.0467 <sup>d</sup>	.0089 <sup>d</sup>

<sup>a</sup> Based on samples dried at 105 °C for one hour. Moisture loss: for SRM 113a 0.08%; for SRM 329 0.4%.

<sup>b</sup> Figures in parenthesis are not certified but are given for information only.

<sup>c</sup> Not determined.

<sup>d</sup> Troy ounces per ton is 13.63 for SRM 113a and 2.60 for SRM 329.

**NOTE:** The total of constituents reported for SRM 113a is 97.6% and that for SRM 329 is 98.0%. The remainder is expected to consist mainly of oxygen, carbon, and water of crystallization.

**CERTIFICATION:** The value listed for a certified element is the present best estimate of the "true" value based on the results of the analytical program. The value listed is not expected to deviate from the "true" value by more than  $\pm 1$  in the last significant figure reported. For a subscript figure, the deviation is not expected to be more than  $\pm 5$ . Based on the results of homogeneity testing, maximum variations within and among samples are estimated to be less than the uncertainty figures given above.

Washington, D.C. 20234  
 December 29, 1975  
 (Revision of Certificate of May 6, 1974 for  
 change in silver results)

J. Paul Cali, Chief  
 Office of Standard Reference Materials

(over)

The material for these standards was supplied by Cominco American Inc., Spokane, Wash. The material for SRM 113a was prepared at the Magmont Mines, Bixby, Mo., and that for SRM 329 at the Sullivan Mine, Trail, B.C., Canada.

Following sieving and blending operations at NBS, homogeneity testing was performed by S. D. Rasberry and J. McKay, (x-ray fluorescence analyses); by E. J. Maienthal, (polarographic analyses); and by R. K. Bell, (chemical analyses).

Selected samples representative of the lot were analyzed and no significant variability was observed when using subsamples of 0.5 g or larger. (Moisture determinations usually were made on larger samples--up to 10 g.)

Cooperative analyses for certification were performed in the analytical laboratories of Cominco, Sullivan Mine, Trail, B.C., Canada, C. J. Mitchell; Cominco American Inc., Magmont Mines, Bixby, Mo., R. J. Gibson; and St. Joe Minerals Corp., Zinc Smelting Division, Monaca, Pa., J. J. Aldrich.

Analyses were performed in the NBS Analytical Chemistry Division by R. K. Bell and E. J. Maienthal.

The overall direction and coordination of the technical measurements at NBS leading to certification were performed under the direction of O. Menis and J. I. Shultz.

The technical and support aspects involved in the preparation, certification, and issuance of these SRM's were coordinated through the Office of Standard Reference Materials by R. E. Michaelis.



# National Bureau of Standards

## Certificate of Analysis

### Standard Reference Material 120b

#### Phosphate Rock

#### (Florida)

This standard is a finely powdered material intended for use in checking chemical methods of analysis and in calibration with optical emission and x-ray spectrometric methods of analysis.

See ADDENDUM\* (Over) for Uranium (Radium and Thorium)  
(All results are based on samples dried for 1 hour at 105 °C.)

Percent by Weight

ANALYST*	P <sub>2</sub> O <sub>5</sub>	CaO	SiO <sub>2</sub>	F	Soluble Fe <sub>2</sub> O <sub>3</sub>	Soluble Al <sub>2</sub> O <sub>3</sub>	MgO	Na <sub>2</sub> O	MnO	K <sub>2</sub> O	TiO <sub>2</sub>	CO <sub>2</sub>	CdO
1	34.51 <sup>a</sup>	49.42 <sup>b</sup>	4.70 <sup>c</sup>	3.82 <sup>d</sup>	1.10 <sup>e</sup>	1.09 <sup>f</sup>	0.29 <sup>g</sup>	0.33 <sup>f</sup>	0.032 <sup>h</sup>	0.12 <sup>fj</sup>	..	0.15 <sup>k</sup>	0.002 <sup>l</sup>
2	34.51 <sup>m</sup>	49.35 <sup>m</sup>	4.73 <sup>n</sup>	3.79 <sup>m</sup>	1.10 <sup>h</sup>	1.07 <sup>h</sup>	.28 <sup>h</sup>	.36 <sup>h</sup>	.031 <sup>h</sup>	.12 <sup>j</sup>	0.09 <sup>o</sup>	..	2.76 <sup>p</sup> .002 <sup>h</sup>
3	34.66 <sup>n</sup>	49.38 <sup>m</sup>	4.67 <sup>q</sup>	3.83	1.09 <sup>h</sup>	1.07 <sup>h</sup>	.30	.36 <sup>h</sup>	.032 <sup>h</sup>	.12 <sup>j</sup>	.098 <sup>o</sup>	.15	2.79 .002 <sup>h</sup>
4	34.67 <sup>r</sup>	49.47 <sup>m</sup>	4.69 <sup>q</sup>	3.81 <sup>s</sup>	1.13 <sup>h</sup>	1.04 <sup>h</sup>	.28 <sup>h</sup>	.35 <sup>h</sup>	.032 <sup>h</sup>	..	.087 <sup>o</sup>	.15 <sup>k</sup>	2.78 <sup>p</sup> .003 <sup>h</sup>
5	34.57	49.32 <sup>m</sup>	4.63 <sup>q</sup>	3.86	1.06 <sup>h</sup>	1.05 <sup>h</sup>	.25 <sup>h</sup>	.34 <sup>h</sup>	..	..	.085 <sup>o</sup>	..	2.83 ..
6	34.48 <sup>m</sup>	49.45 <sup>m</sup>	..	3.92 <sup>s</sup>	1.14 <sup>m</sup>	1.07 <sup>t</sup>	..	..	..	..	..	..	..
Average	34.57	49.40	4.68	3.84	1.10	1.06	0.28	0.35	0.032	0.12	0.090	0.15	2.79 0.002

<sup>a</sup> Phosphorus precipitated with magnesia mixture, ignited and weighed as Mg<sub>2</sub>P<sub>2</sub>O<sub>7</sub>.

<sup>b</sup> Calcium precipitated as oxalate, ignited and weighed as CaO.

<sup>c</sup> Sample fused with Na<sub>2</sub>CO<sub>3</sub>, silica precipitated with ZnO and dehydrated with HCl. Traces of SiO<sub>2</sub> recovered by H<sub>2</sub>SO<sub>4</sub> dehydration.

<sup>d</sup> Fluorine distilled into NaOH solution and precipitated as lead chlorofluoride. Chloride is precipitated with excess AgNO<sub>3</sub> and excess AgNO<sub>3</sub> is titrated with standard KCNS solution.

<sup>e</sup> SnCl<sub>2</sub> reduction - K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> titration.

<sup>f</sup> Flame emission spectrometry with repetitive optical scanning.

<sup>g</sup> A value of 1.13 percent was obtained for total Al<sub>2</sub>O<sub>3</sub> by gravimetry.

<sup>h</sup> Atomic absorption spectrometry.

<sup>i</sup> KIO<sub>4</sub> spectrophotometric method.

<sup>j</sup> Sample digested with mixed acids for 1 hour. Determination completed by atomic absorption spectrometry.

<sup>k</sup> H<sub>2</sub>O<sub>2</sub> spectrophotometric method.

<sup>l</sup> Polarographic method.

<sup>m</sup> Volumetric method.

<sup>n</sup> Gravimetric method.

<sup>o</sup> Sample digested with dilute HCl or aqua regia for 15 minutes. Determination completed by atomic absorption spectrometry.

<sup>p</sup> CO<sub>2</sub> absorbed and weighed.

<sup>q</sup> Dehydration with HClO<sub>4</sub> in presence of boric acid.

<sup>r</sup> Molybdovanadophosphate spectrophotometric method.

<sup>s</sup> Distillation - titration with standard thorium nitrate solution.

<sup>t</sup> Aluminum precipitated with 8 hydroxyquinoline and weighed.

Washington, D.C. 20234  
July 31, 1972  
ADDENDUM\* (Over)  
July 31, 1979

J. Paul Cali, Chief  
Office of Standard Reference Materials

(over)

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of O. Menis and J. I. Shultz.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. E. Michaelis and C. L. Stanley.

**PREPARATION, TESTING, AND ANALYSIS:** The material for this standard was prepared by the American Cyanamid Company. Eighty five percent of the lot was made to pass 200 mesh sieve and some blending was done at the plant. Final sieving and blending operations were accomplished at NBS.

Homogeneity testing was performed by S. D. Rasberry, C. E. Fiori, and J. McKay with x-ray fluorescence analysis. Calcium and phosphorus determinations were made on 14 samples representative of the top and the bottom of seven containers. The size of the samples taken for analysis was approximately 35 mg. The maximum variations in concentration among samples were within 0.09 percent for CaO and 0.12 percent for  $P_2O_5$ .

The laboratories and analysts cooperating in the analytical program for certification were:

1. R. K. Bell, E. R. Deardorff, E. J. Maienthal, T. C. Rains, T. A. Rush, and S. A. Wicks, Analytical Chemistry Division, Institute for Materials Research, National Bureau of Standards.
2. J. Padar, Agrico Chemical Co., Division of Continental Oil Company, Pierce, Florida.
3. D. B. Underhill, Borden Chemical Co., Plant City, Florida.
4. C. C. Thornton, Thornton Laboratories, Inc., Tampa, Florida.
5. W. W. Harwood, R. M. Lynch and H. N. Gomez, International Minerals and Chemical Corp., Bartow, Florida.
6. J. A. Sielski, American Cyanamid Co., Brewster Plant, Bradley, Florida.

#### \*ADDENDUM

Uranium has been determined at NBS by thermal ionization mass spectrometry, E. L. Garner and L. A. Machlan, and the following certification is made:

	Value, $\mu\text{g/g}$	Estimated Uncertainty <sup>a</sup>
Uranium	128.4	$\pm 0.5$

<sup>a</sup>The estimated uncertainty is based on judgment and represents an evaluation of method imprecision and material variability.

(NOTE: On similar phosphate rock materials, a value of 127  $\mu\text{g/g}$  for uranium was reported in Ref. 1; additionally, values of 17  $\mu\text{g/g}$  for thorium and 43  $\text{pCi}^{225}\text{Ra/g}$  for radium also were reported.)

Ref. 1 Agr. Food Chem., 16, No. 2, 1968 (p232)

U. S. Department of Commerce  
Frederick B. Dent  
Secretary

National Bureau of Standards  
Richard W. Roberts, Director

# National Bureau of Standards

## Certificate of Analysis

### Standard Reference Material 154b

#### Titanium Dioxide

This standard is in the form of fine powder, certified primarily for application in the paint and ceramic industries.

<u>Constituent</u>	<u>Percent by Weight</u> <sup>a</sup>	<u>Uncertainty</u> <sup>c</sup>
Titanium Dioxide (TiO <sub>2</sub> )	99.74 <sup>b</sup>	0.05

<sup>a</sup> Based on material dried at 110 °C for two hours.

<sup>b</sup> The value given in this certificate is based on the following pertinent analytical data:

<sup>c</sup> The uncertainty figure represents the 95% confidence interval of the mean of all accepted values.

<u>Method</u>	<u>NBS</u>		<u>Number of Determinations</u>
	<u>Average</u>	<u>Standard Deviation</u> <sup>1</sup>	
Controlled-potential coulometric (0.2g samples)	99.73	0.05	9
Volumetric (0.35g samples)	99.71	0.03	10 <sup>2</sup>
<u>Cooperators</u> <sup>3</sup>			
Volumetric (Analyst A) <sup>4</sup>	99.75	0.04	3
Volumetric (ASTM D1394)	99.78	0.05	3

<sup>1</sup> Of single determinations.

<sup>2</sup> Two discrepant results were omitted.

<sup>3</sup> Results from one cooperating laboratory were deemed significantly high and have been omitted.

<sup>4</sup> Results from Analyst B at the same laboratory were deemed significantly low and have been omitted.

Washington, D. C. 20234  
May 16, 1973

J. Paul Cali, Chief  
Office of Standard Reference Materials



PLANNING, PREPARATION, TESTING, and ANALYSIS: The material for this SRM has been carefully selected and prepared not only to reflect the present composition needs but also the anticipated future requirements.

A particular ilmenite ore was chosen so that, after beneficiation, the material would provide the desired high rutile to anatase ratio (rutile 97+%, anatase about 2%). Bleaching agents were not added; thus the material has the characteristically yellowish color of rutile. The lot was thoroughly blended in the laboratory and then screened through a 44 $\mu$ m (325 mesh) sieve. Preliminary testing on 6 samples, representative of the lot, showed no evidence of inhomogeneity. The planning, preparation and preliminary testing were under the supervision of John J. Libera, Research and Development Department, National Lead Industries, St. Louis, Missouri.

Cooperative analyses for certification were performed in the analytical laboratories of E. I. Du Pont De Nemours & Co., Pigments Department, Wilmington, Delaware, T. D. McKinley; National Lead Industries, Titanium Pigment Division, South Amboy, New Jersey, Benjamin S. Sanderson; and Sherwin-Williams Research Center, Chicago, Illinois, R. W. Scott.

Analyses were performed in the Analytical Chemistry Division of the National Bureau of Standards by J. R. Baldwin and G. Marinenko.

The overall direction and coordination of the technical measurements at NBS leading to certification were performed under the direction of O. Menis and J. I. Shultz.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. E. Michaelis and C. L. Stanley.

#### ADDITIONAL INFORMATION ON THE COMPOSITION:

Certification is made only for the TiO<sub>2</sub> content. Investigations of this material at NBS and at cooperating laboratories provided some additional information that may be useful, but is not certified:

Constituent	Weight Percent
P <sub>2</sub> O <sub>5</sub>	(0.04)
SiO <sub>2</sub>	(0.01)
Fe <sub>2</sub> O <sub>3</sub>	(0.006)
Pb	(0.003)
CaO	(~0.01)
V	(~0.001)
Cr	(~0.0005)
Cu	(~0.0005)
MgO	(~0.01)
Moisture (110 °C-2 hours)	(0.02 to 0.05)
Loss on Ignition (900 °C for one hour under helium)	(0.06 to 0.07)

If in the use of this SRM, determinations are made for any of the uncertified minor and trace constituents, it would be appreciated if the results were forwarded to the Office of Standard Reference Materials. When sufficient information has been received the Certificate will be revised.



U.S. Department of Commerce  
Juanita M. Krep  
Secretary

National Bureau of Standards  
Ernest Ambler, Director

# National Bureau of Standards

## Certificate of Analysis

### Standard Reference Material 165a

#### Glass Sand

(In Cooperation with the American Society for Testing and Materials)

This SRM has been blended to ensure homogeneity. It should be dried for 2 hours at 105 °C before use.

<u>Constituent</u>	<u>Percent by Weight</u>	<u>Range</u>	<u>s</u>
Al <sub>2</sub> O <sub>3</sub>	0.059	0.051 - 0.066	0.0024
Fe <sub>2</sub> O <sub>3</sub>	.012	.007 - .017	.0018
TiO <sub>2</sub>	.011	.0065 - .015	.0016
ZrO <sub>2</sub>	.006	.0005 - .012	.002

Certification - The value listed for each oxide is the best estimate of the true value based on the analytical data from both cooperators and NBS. The range of values listed is the tolerance interval, constructed such that it will cover at least 95% of the population with a probability of 0.99. It is computed as  $\bar{X} \pm Ks$ : where  $s$  is the standard deviation,  $K$  is a factor that depends on  $n$  (the number of samples measured),  $p$ , the proportion of the total samples covered (95%), and  $\gamma$ , the probability level (99%). In all cases none of the  $n$  values used exceeded the range specified. Thus, it includes variability between laboratories and samples.

The overall direction and coordination of the round-robin analysis leading to certification were performed by Paul Close, Chairman of ASTM Subcommittee C-14.02 on Chemical Analysis of Glass and Glass Products.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by W. P. Reed.

Washington, D.C. 20234  
October 16, 1978

J. Paul Cali, Chief  
Office of Standard Reference Materials

(over)

#### Additional Information

A content of 1  $\mu\text{g/g}$  for  $\text{Cr}_2\text{O}_3$  is not certified but rather is provided for information only.

Chemical analyses for certification were performed in the following laboratories:

Anchor Hocking Corp., Lancaster, Ohio, R. E. Carr.

Brockway Glass Co., Inc., Brockway, Pa., E. L. McKinley.

Corning Glass Works, Corning, N.Y., Y. S. Su.

Ford Motor Co., Lincoln Park, Mich., T. L. LaFramboise.

National Bureau of Standards, Analytical Chemistry Division, E. J. Maienthal, J. D. Messman and T. C. Rains.

Kimble Div. Owens-Illinois, Vineland, N.J., H. S. Moser.

Owens-Illinois, Inc., Toledo, Ohio, P. Close.

Penn State Univ., University Park, Pa., J. B. Bodkin.

# Certificate of Analysis

## Standard Reference Material 180

### High-Grade Fluorspar

This Standard Reference Material has a high calcium fluoride content and is issued primarily for the geological and geochemical scientific community. [NOTE: This material is not a substitute for the fluorspar standard, SRM 79, used primarily for the assay of fluorspar imported for industrial use.]

<u>Constituent</u>	<u>Percent, by weight</u>
CaF <sub>2</sub>	98.80 <sup>a</sup> ± 0.03 <sup>b</sup>

<sup>a</sup>The value certified is based entirely on the method given in this certificate and is the mean of eight determinations.

<sup>b</sup>The standard deviation of a single determination.

Trace elements: These were determined by a general qualitative spectrochemical method and are given for information only. Fe, 0.1 – 1.0%; Al, Ba, Mg, Pb, Si, and Sr, 0.01 – 0.1%; Cu, K, Mn, Na, Ti, and V, 0.001 – 0.01%; and Ag and Li, less than 0.001%. Analyst - E. K. Hubbard.

The analytical work leading to certification was performed by K. M. Sappenfield, Analytical Chemistry Division, National Bureau of Standards.

This material was supplied by Minera Frisco of San Francisco Del Oro, Chih., Mexico.

The technical and support aspects involved in the preparation, certification, and issuance of this standard reference material were coordinated through the Office of Standard Reference Materials by J. L. Hague, R. E. Michaelis, and C. L. Stanley.

Washington, D. C. 20234  
March 31, 1971

J. Paul Cali, Chief  
Office of Standard Reference Materials

(over)

## METHOD FOR THE DETERMINATION OF $\text{CaF}_2$ IN FLUORSPAR

\*Transfer 0.50 g of fluorspar, previously dried at 100 to 105 °C, to a tared, ignited platinum crucible. Add 15 ml of acetic acid (1:9) containing 0.667 g of  $\text{CaCO}_3$  per liter. Digest on a steam bath for 30 minutes and stir at five minute intervals. Add about 0.04 g of ashless filter pulp and stir for an additional minute. Filter through a double 12.5 cm extra dense filter paper (S & S No. 589 Red Ribbon) to which has been added about 0.04 g ashless filter pulp. Thoroughly wash the crucible, paper, and stirring rod with 5 ml portions of hot water (40 to 60 °C), using a total of about 35 ml. After washing, wipe the stirring rod with a small piece of wet filter paper and add the paper to the filter. Transfer the paper and residue to the crucible, dry in an oven at 80 °C, char slowly under an infrared lamp, and ignite in a muffle furnace at 600 °C.

\*\*Add 3 ml of HF to the residue in the crucible and evaporate to dryness. Add 1 to 2 ml of  $\text{HClO}_4$  and evaporate to dryness under the hood. Cool, wash the inside of the crucible with 1 ml more of  $\text{HClO}_4$  (to catch any undecomposed particles of calcium fluoride), and again evaporate to dryness. Cool the crucible and immerse in a beaker containing 150 ml of dilute HCl (5:95). Warm the beaker gently and remove the platinum crucible, being sure to remove adhering particles of  $\text{Ca}(\text{ClO}_4)_2$ .

Boil the contents of the beaker for 10 minutes. If any insoluble matter remains; filter, wash, and ignite it in platinum. Treat the ignited residue with a few drops of HF and  $\text{HClO}_4$  and heat to expel the acids. Add 1 to 2 ml of HCl and digest on a steam bath. Transfer the contents to the main filtrate.

Pass  $\text{H}_2\text{S}$  into the clear filtrate for several minutes and then make the solution ammoniacal. Continue with  $\text{H}_2\text{S}$  for 10 minutes and allow the precipitate to settle for 20 to 30 minutes. Filter and wash with  $\text{NH}_4\text{Cl}-(\text{NH}_4)_2\text{S}$  solution. (Prepare by passing a moderate stream of  $\text{H}_2\text{S}$  for 5 minutes through a solution containing 5 ml of  $\text{NH}_4\text{OH}$  and 10 g of  $\text{NH}_4\text{Cl}$  per liter.)

Neutralize the filtrate and washings with HCl, and add an additional 20 ml of HCL. Boil for 2 to 3 minutes to expel most of the  $\text{H}_2\text{S}$ . Add KBr- $\text{Br}_2$  solution (20% solution of KBr saturated with  $\text{Br}_2$ ) until the solution remains yellow. Boil until the finely divided sulfur has been oxidized and the bromine has been expelled. If the sulfur or sulfides are not removed by the bromine treatment, filter, wash the paper well and discard the residue. Dilute the filtrate to about 200 ml. Precipitate the calcium by adding 2 g of  $(\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$  and, while stirring, slowly add  $\text{NH}_4\text{OH}$  until the solution is slightly ammoniacal. Heat on a steam bath for one-half to one hour, stirring occasionally. Cool to room temperature. Filter on a close textured paper. Wash with a cold 0.1 percent solution of  $(\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$ . Ignite to constant weight. Calculate the percent  $\text{CaF}_2$  from the weight of the  $\text{CaO}$ .

### References:

\*Removal of soluble calcium in acetic acid. Interlaboratory study of soluble calcium.

\*\*Method: Calcium in fluorspar

The Analysis of Fluorspar by G. E. F. Lundell and J. I. Hoffman, J. Res. Nat. Bur. Stand. (U.S.), 2 (1929) R.P. 51.

**Certificate of Analysis**  
**STANDARD REFERENCE MATERIALS**  
**181, 182, and 183**  
**Lithium Ores**

These SRM's are intended for use in checking the accuracy of assay methods. They are certified for their constituent of economic interest. Additional data for information only appears on page 2. These SRM's are supplied in the form of fine powder.

	SRM 181 (Spodumene) %	SRM 182 (Petalite) %	SRM 183 (Lepidolite) %
Li <sub>2</sub> O	6.3 <sub>9</sub>	4.3 <sub>4</sub>	4.1 <sub>2</sub>

The value listed for Li<sub>2</sub>O in the three SRM's is the best estimate of the "true" value. The deviation is not expected to be more than  $\pm 5$  in the subscript number.

Washington, D.C. 20234  
October 1, 1981  
(Revision of Certificate dated  
2/24/58 and Reprinted 8/20/70)

George A. Uriano, Chief  
Office of Standard Reference Materials

(over)

The following values are approximate, and are listed only for information.

<u>Constituent</u>	<u>Wt. %</u>	<u>Wt. %</u>	<u>Wt. %</u>
Na <sub>2</sub> O	(0.8)	(0.4)	(0.2)
K <sub>2</sub> O	(.3)	(.1)	(8. )
Rb <sub>2</sub> O	- - -	(.03)	(3.5)
Cs <sub>2</sub> O	- - -	- - -	(.3)

UNITED STATES DEPARTMENT OF COMMERCE  
WASHINGTON 25, D.C.

National Bureau of Standards  
Certificate of Analyses  
Standard Sample 198  
Silica Brick

(All results are based on samples dried at 105° to 110° C.)

Analyst	Al <sub>2</sub> O <sub>3</sub>	Total iron as Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	ZrO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	MnO	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	Li <sub>2</sub> O	*Loss on ignition
1.....	{ b 0.17 + 0.15 b 19 b 64	{ + 0.65 + 0.67 b 01 b 64	0.02	< 0.01	b 0.022 b 012	+ 0.006	2.72	0.07	+ 0.007 b 009	+ 0.016 b 010	+ 0.001 b 001	0.21 .32
2.....	{ b 17 + 15 b 15	{ + 0.67 + 0.65 + 69	.01		+ 0.011		2.75	.06	+ 0.02	+ 0.03		.22
3.....	b 17	+ 0.69	.02		+ 0.026		2.73	.08	+ 0.008	b 016	b < .007	.15
4.....	b 17	+ 0.66	.01		+ 0.025		2.72	.07	+ 0.005	+ 0.012	+ 0.002	.23
5.....		.68	.03		.026		2.72	.07	+ 0.005		+ 0.001	.23
6.....	b 16	+ 0.68	.03		.025	< .005	2.69	.09	+ 0.02	+ 0.02	+ 0.001	.22
7.....	b 16	+ 0.66	.02		b 019		2.70	.08				.20
8.....	b 15	+ 0.66	.02		{ b 024 b 02	+ 0.008	2.74	.07			b < .01	.14
9.....	b 16	+ 0.66	.01	nil			2.67	.06				.20
10.....	+ 0.16	+ 0.68	.01		+ 0.024	+ 0.01	2.67	.09				
11.....	b 16	+ 0.67	.02		+ 0.024	.008	2.71	.08	+ 0.02	+ 0.018		
Average.....	0.16	0.66	0.02		0.022	0.008	2.71	0.07	0.012	0.017	0.001	0.21

\* 1 g heated at 900° to 1,000° C. in a covered platinum crucible to constant weight.  
b Weighed United NH<sub>4</sub>OH precipitate corrected for Fe<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub>, and P<sub>2</sub>O<sub>5</sub>.  
c Aluminum separated from iron, titanium, etc., with sodium hydroxide, precipitated, and weighed as AlPO<sub>4</sub>.  
d Same value obtained by the aluminum photometric method.  
e Thiocyanate photometric method.  
f NaCl-K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> method.  
g Same value obtained gravimetrically as Fe<sub>2</sub>O<sub>3</sub>.  
h Molybdenum-blue photometric method.

i Periodate photometric method.  
j Flame-photometric method.  
k Spectrographic determination.  
l Aluminum separated from iron and titanium by ion exchange, and weighed as aluminum oxyquinolate.  
m NaCl-K<sub>2</sub>MnO<sub>4</sub> method.  
n Iron separated from aluminum and titanium by ion exchange, precipitated with ammonium hydroxide, and weighed as Fe<sub>2</sub>O<sub>3</sub>.  
o Gravimetric. Weighed as Mg<sub>2</sub>P<sub>2</sub>O<sub>7</sub>.  
p Titration with AgNO<sub>3</sub> following ion exchange separation of sodium and potassium chlorides.

q 6-Hydroxyquinoline precipitation. Bromate-thiosulfate titration. See Trans. British Ceramic Society 51, No. 9, 438 (1952).  
r Sulfuric acid-sulfate-gravimetric method.  
s Titrated with Ti(SO<sub>4</sub>)<sub>2</sub>.  
t Phosphomolybdate-alkalimetric method.  
u Weighed as AlPO<sub>4</sub>.  
v Orthophosphoric acid photometric method.  
w Same value obtained by microchromatography-R photometric method.  
x Ferriate-cerise method.

List of Analysts

1. K. M. Sappenfield and R. A. Paulson, National Bureau of Standards.
2. G. R. Eusner, U. S. Steel Corp., Monroeville, Pa.
3. B. E. Gurnio, W. O. Osborn, and R. N. Smith, Crescent Division, North American Refractories Co., Curwensville, Pa.
4. M. P. Bennett and Faiba Whitney, Gladding, McBean & Co., Los Angeles, Calif.
5. B. C. Ruprecht, Harbison-Walker Refractories Co., Pittsburgh, Pa.

6. A. R. Lesar, A. P. Green Fire Brick Co., Mexico, Mo.
7. L. J. Trostel, General Refractories Co., Baltimore, Md.
8. D. J. Halliscy, Jones & Laughlin Steel Corp., Pittsburgh, Pa.
9. J. J. Hazel, Republic Steel Corp., Cleveland, Ohio.
10. Paul Smith, The Refractories Institute, Mellon Institute, Pittsburgh, Pa.
11. J. B. Armstrong, Sparrows Point Plant, Bethlehem Steel Co., Sparrows Point, Md.
12. W. F. Zollinger, Bethlehem Steel Co., Bethlehem, Pa.

WASHINGTON 25, D.C., January 6, 1960.

U. S. GOVERNMENT PRINTING OFFICE 337563

A. V. ASTIN, Director.

UNITED STATES DEPARTMENT OF COMMERCE  
WASHINGTON 25, D.C.

**National Bureau of Standards**  
**Certificate of Analyses**  
**Standard Sample 199**  
**Silica Brick**

(All results are based on samples dried at 105° to 110° C.)

Analyst	Al <sub>2</sub> O <sub>3</sub>	Total iron as Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	ZrO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	MnO	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	Li <sub>2</sub> O	* Loss on ignition
1.....	<sup>b</sup> 0.47 " 48	<sup>d</sup> 0.76 " 74	0.07	0.01	<sup>f</sup> 0.010	" 0.005	2.41	0.13	<sup>h</sup> 0.015	<sup>h</sup> 0.09	<sup>h</sup> 0.001	0.14
2.....	1.49	1.70	1.05		1.016		<sup>i</sup> 2.45	<sup>i</sup> .14	1.010	<sup>i</sup> .07	<sup>i</sup> .002	.23
3.....	<sup>b</sup> 1.48	" 75	.05				2.42	.12	1.02	<sup>i</sup> .11		.16
4.....	" 48	" 73	.06		" 026		2.38	.14	" 014	<sup>h</sup> .11	<sup>i</sup> < .007	.12
5.....	<sup>b</sup> .51	" 74	.06		" 012		2.39	.13	<sup>h</sup> 0.010	<sup>h</sup> .088	<sup>h</sup> .002	.16
6.....			.07		.015	.008	2.46	.14	<sup>h</sup> .004	<sup>h</sup> .10	<sup>h</sup> .001	.12
7.....	<sup>b</sup> .50	" 75	.07		.025	< .005	2.43	.10	<sup>h</sup> .02	<sup>h</sup> .09	<sup>h</sup> .002	.16
8.....	" 48	" 76	.07		<sup>f</sup> .005		2.38	.14				.13
9.....	" 72	" 76	.06		" 013	" 007		<sup>i</sup> .14			<sup>i</sup> < .01	.30
10.....	<sup>b</sup> .49	" 75	.06	nil	.03		2.37	.13				.17
11.....	" 1.48	" 74	.06		" 008	" 01	2.38	.16		" 097		
12.....	" 48	" 74	.06		" 010	" 007	2.39	.14	<sup>h</sup> .03	<sup>h</sup> .087		
Average.....	0.48	0.74	0.06		0.015	0.007	2.41	0.13	0.015	0.094	0.002	0.17

\* 1 heated at 900° C. to 1,000° C. in a covered platinum crucible to constant weight.  
<sup>b</sup> Weighed ignited NH<sub>4</sub>OH precipitate corrected for ZrO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub>, and P<sub>2</sub>O<sub>5</sub>.  
<sup>c</sup> Aluminum separated from iron, titanium, and zirconium by cupferron, precipitated with ammonium hydroxide, weighed as Al(OH)<sub>3</sub> and corrected for P<sub>2</sub>O<sub>5</sub>.  
<sup>d</sup> SiO<sub>2</sub>-K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> method.  
<sup>e</sup> Weighed as Fe<sub>2</sub>O<sub>3</sub>.  
<sup>f</sup> Molybdenum-blue photometric method.  
<sup>g</sup> Periodate photometric method.

<sup>h</sup> Flame-photometric method.  
<sup>i</sup> Spectrographic determination.  
<sup>j</sup> Same value obtained by separating aluminum from iron and titanium by ion exchange, and weighing as aluminum oxyquinolate.  
<sup>k</sup> SiO<sub>2</sub>-K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> method.  
<sup>l</sup> Titration with AgNO<sub>3</sub> following ion exchange separation of sodium and potassium chlorides.  
<sup>m</sup> 6-Hydroxyquinoline precipitation. Bromate-thiourea titration. See Trans. British Ceramic Society 51, No. 9, 438 (1952).

\* Gravimetric. Weighed as Mg<sub>2</sub>P<sub>2</sub>O<sub>7</sub>.  
<sup>h</sup> Sodium uranyl zinc acetate-gravimetric method.  
<sup>i</sup> Titrated with Ti(SO<sub>4</sub>)<sub>2</sub>.  
<sup>j</sup> Phosphomolybdate-spectrometric method.  
<sup>k</sup> Weighed as AlPO<sub>4</sub>.  
<sup>l</sup> Orthophosphatocolorimetric method.  
<sup>m</sup> Same value obtained by eriochrome cyanine-R photometric method.  
<sup>n</sup> Permanganate-arsenite method.  
<sup>o</sup> Gravimetric. Decomposition with NH<sub>4</sub>Cl-CaCO<sub>3</sub>.

List of Analysts

1. K. M. Sappenfield and R. A. Paulson, National Bureau of Standards.
2. G. R. Eusner, U.S. Steel Corp., Monroeville, Pa.
3. B. E. Gummo, W. O. Osborn, and R. N. Smith, Crescent Division, North American Refractories Co., Curwensville, Pa.
4. M. P. Bennett and Falba Whitney, Gladding, McBean & Co., Los Angeles, Calif.
5. B. C. Ruprecht, Harbison-Walker Refractories Co., Pittsburgh, Pa.
6. A. R. Lesar, A. P. Green Fire Brick Co., Mexico, Mo.
7. L. J. Trostel, General Refractories Co., Baltimore, Md.
8. D. J. Hallisey, Jones & Laughlin Steel Corp., Pittsburgh, Pa.
9. J. J. Hazel, Republic Steel Corp., Cleveland, Ohio.
10. Paul Smith, The Refractories Institute, Mellon Institute, Pittsburgh, Pa.
11. J. B. Armstrong, Sparrows Point Plant, Bethlehem Steel Co., Sparrows Point, Md.
12. W. F. Zollinger, Bethlehem Steel Co., Bethlehem, Pa.

A. V. ASTIN, Director.

WASHINGTON 25, D.C., January 6, 1960.

U.S. GOVERNMENT PRINTING OFFICE 1375483



# National Bureau of Standards

## Certificate of Analysis

### Standard Reference Material 277

#### Tungsten Concentrate

(In cooperation with the American Society for Testing and Materials)

This material is in the form of powder ( $<0.15$  mm) intended for use in checking chemical methods of analysis and in calibration with instrumental methods of analysis.

Constituent	Certified Value <sup>1</sup> (wt%)	Estimated Uncertainty <sup>2</sup> (wt%)
WO <sub>3</sub>	67.4	0.3

(Results are based on samples dried at 110 °C for one hour)

- <sup>1</sup> The certified value is the *present best estimate* of the "true" value based on the results of the cooperative program for certification.
- <sup>2</sup> The estimated uncertainty is based on judgment and represents an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material variability for samples of 1 g or more (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determination.)

CAUTION: The material for this SRM primarily was derived from wolframite ores. It is, however, a mixture of concentrates from China, Thailand, and USA that contains titanium, niobium, tantalum, and tin in amounts not normally encountered in most wolframite concentrates. These constituents may interfere in the "classic" chemical procedures and may necessitate appropriate changes in methodology. Also, x-ray fluorescence methods of analysis that use SRM 277 to make relative measurements of "pure" wolframite (or scheelite) concentrates may exhibit systematic errors because of the unusual constituents contained in this concentrate.

The overall coordination of the technical measurements leading to certification were performed under the direction of J. I. Shultz, Research Associate, NBS-ASTM Research Associate Program.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. E. Michaelis.

Washington, D.C. 20234  
October 24, 1978

J. Paul Cali, Chief  
Office of Standard Reference Materials

(over)

PLANNING, PREPARATION, TESTING, ANALYSIS: The material for SRM 277 was carefully selected and provided to NBS by GTE Sylvania, Towanda, Pennsylvania, courtesy of J. Demangone. At Sylvania, the material was crushed and ground to a fine powder. At NBS the material was sieved ( $<0.15$  mm) and thoroughly blended.

Homogeneity testing of selected samples representative of the lot of SRM 277 was performed by an x-ray fluorescence technique by R. E. Michaelis. The material variability was determined to be within  $\pm 0.1$  (wt%) of  $WO_3$ .

Cooperative analyses for certification were performed in the following analytical laboratories:

Alfred H. Knight International, Ltd., Cheshire, U.K. - J. F. L. Knight.

Benedict Kitto and Sons, London, U.K. - R. Peck.

General Electric Company, Cleveland, Ohio - J. Rynasiewicz and J. W. Fulton.

GTE Sylvania, Towanda, Pa. - R. Dyck; and Waltham, Mass. - J. F. Cosgrove.

Hermann C. Starck, Berlin, Germany - O. Hilmer.

Ledoux & Company, Teaneck, N. J. - S. Kallmann.

National Bureau of Standards, Washington, D.C. - E. R. Deardorff.

Sandvik, A. B., Stockholm, Sweden - K. Kåarik; and Sandvik Asia Ltd. - N. R. Sanjana.

Spectro Chem Labs, Inc., Franklin Lakes, N. J. - E. W. Hobart.

Treibacher Chemische Werke, Treibach, Austria - Z. Otto.

Westinghouse Electric Corporation, Bloomfield, N. J. - P. J. Walitsky.

Union Carbide Corporation, Bishop, Calif. - E. C. Gibbs and K. M. Wilder; and Niagara Falls, N. Y. - P. Greenberg.

VEW Ternitz, Austria - A. J. Leeb.

NOTE: Details regarding the methodology employed in the analysis of this SRM, along with other pertinent information, will appear in a separate publication.

ADDITIONAL INFORMATION OF THE COMPOSITION: Certification is made only for the  $WO_3$  content; however, SRM 277 contains additional constituents of interest as indicated below. These are *not certified* and are provided for information only. Table 1 lists those constituents for which data were received from two or more laboratories that were in good agreement. (Most of these are expected to be proposed for certification at a later date and, therefore, an indication of the uncertainty also is given.) Table 2 lists those constituents for which data were received from a single laboratory (or discrepant data from two or more laboratories).

NOT CERTIFIED

Table 1

<u>Constituent</u>	<u>Approximate Value (wt%)</u>	<u>Approximate Uncertainty (wt%)</u>
Calcium	(0.37)	(0.02)
Iron	(7.4)	(.1)
Lead	(0.07)	(.01)
Manganese	(10.0)	(.2)
Molybdenum	(0.06)	(.01)
Niobium	(1.00)	(.03)
Phosphorus	(0.03)	(.01)
Silicon	(.85)	(.05)
Sulfur	(.25)	(.03)
Tin	(.54)	(.07)
Titanium	(2.2)	(.2)

NOT CERTIFIED

Table 2

<u>Constituent</u>	<u>Information Value (wt%)</u>	<u>Constituent</u>	<u>Information Value (wt%)</u>
Arsenic	(0.015)	Oxygen	(21.4)
Bismuth	(.07)	Scandium	(0.05)
Cerium	(.03)	Tantalum	(.20)
Chromium	(.04)	Thorium	(.08)
Copper	(.02)	Uranium	(.13)
Gadolinium	(.01)	Yttrium	(.03)
Lanthanum	(.02)	Zirconium	(.01)
Neodymium	(.03)		

In addition to the above, trace concentrations were reported (ppm by wt.) for the following as an indication of the relative amounts observed: Antimony (3), Barium (20), Boron (20), Dysprosium (30), Erbium (20), Europium (10), Holmium (5), Hafnium (20), Indium (20), Lutetium (5), Praseodymium (50), Samarium (40), Tellurium (2), Terbium (10), Thallium (2), Thulium (10), Vanadium (70), Ytterbium (50) and Zinc (20).

NOTE: Although SRM 277, Tungsten Concentrate, is expected to be stable under normal storage conditions, prudent cautions in the laboratory should be observed. The bottle should be kept tightly capped except when in direct use. Store in a desiccator over desiccant.

# National Bureau of Standards

## Certificate

### Standard Reference Material 278 Obsidian Rock

This Standard Reference Material (SRM) is intended for use in evaluating the accuracy of analytical methods and instruments used in the analysis of geological type materials. SRM 278 is a finely powdered obsidian rock, which was obtained from Clear Lake, Newberry Crater, Oregon.

#### Certified Values of Constituents

The concentrations of the constituents were determined by methods that are widely used in the field of geological analysis and have a demonstrated accuracy. The values given are "certified" values, i.e., those values that were determined by either a definitive method, reference method, or by two or more independent methods, and "information" values that were determined by single or non-reference methods. The certified values are given in Table 1.

Table 1. Certified Values of Constituents

Constituent <sup>1</sup>	Content <sup>2</sup> (wt %)	Constituent <sup>1</sup>	Content <sup>2</sup> wt (μg/g)
Al <sub>2</sub> O <sub>3</sub> <sup>d</sup>	14.15 ± 0.15	Cu <sup>e</sup>	5.9 ± 0.2
CaO <sup>e</sup>	0.983 ± 0.002	Ni <sup>e</sup>	3.6 ± 0.3
FeO <sup>f</sup>	1.36 ± 0.02	Rb <sup>a,e</sup>	127.5 ± 0.3
Fe <sub>2</sub> O <sub>3</sub> <sup>i,f</sup> (Total Fe as Fe <sub>2</sub> O <sub>3</sub> )	2.04 ± 0.02	Sr <sup>e</sup>	63.5 ± 0.1
K <sub>2</sub> O <sup>a,d,e</sup>	4.16 ± 0.02	Th <sup>e,f</sup>	12.4 ± 0.3
MnO <sup>b,f</sup>	0.052 ± 0.002	Tl <sup>e</sup>	0.54 ± 0.04
Na <sub>2</sub> O <sup>a,d,f</sup>	4.84 ± 0.05	U <sup>e</sup>	4.58 ± 0.04
P <sub>2</sub> O <sub>5</sub> <sup>b,d</sup>	0.036 ± 0.003	Pb <sup>e</sup>	16.4 ± 0.2
SiO <sub>2</sub> <sup>d</sup>	73.05 ± 0.13		
TiO <sub>2</sub> <sup>b,g</sup>	0.245 ± 0.007		

<sup>1</sup>Methods of Analysis:

<sup>a</sup>Atomic Absorption

<sup>b</sup>Coulometry

<sup>c</sup>Emission Spectrometry

<sup>d</sup>Gravimetry

<sup>e</sup>Isotope Dilution Mass Spectrometry

<sup>f</sup>Neutron Activation Analysis

<sup>g</sup>Prompt-gamma Activation Analysis

<sup>h</sup>Specific Ion Electrode Potentiometry

<sup>i</sup>Titrimetry

<sup>j</sup>Volumetry

<sup>2</sup>The estimated uncertainties of the certified values are based on judgment and represent an evaluation of the combined effects of method imprecision, possible systematic errors among methods and material variability of 250 mg or more. (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determination of most constituents.)

The overall direction and coordination of the technical measurements leading to certification were performed in the Inorganic Analytical Research Division, E. L. Garner, Chief.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. E. Gills.

Washington, D. C. 20234  
August 19, 1981

George A. Uriano, Chief  
Office of Standard Reference Materials

(over)

## Supplemental Information

### Preparation

The material was processed by the Colorado School of Mines, Golden, Colorado. Approximately 350 lbs of obsidian rock were crushed, ground, and sieved to <200 mesh. The material was mixed in a cone blender to ensure homogeneity. For homogeneity testing and certification samples were randomly chosen and analyzed for both major and minor constituents. The inhomogeneity of this material is considered to be  $\leq 2\%$  relative.

### Analysis

SRM 278, a natural glass, is hygroscopic and contains water that cannot be driven off by drying at low temperatures. This material will pick up additional water on exposure to the atmosphere. Thus, exposure time should be kept to a minimum. Furthermore, the sample should be ignited to a constant weight in a muffle furnace or over a small flame at a temperature between 350-600 °C. This procedure will ensure the accurate and precise determination of SiO<sub>2</sub>, K<sub>2</sub>O, Na<sub>2</sub>O, Al<sub>2</sub>O<sub>3</sub> and possibly other major constituents.

The analysts and laboratories cooperating in the analytical program for certification were:

M. J. Blackman, E. L. Garner, J. W. Gramlich, L. A. Machlan, L. J. Moore and R. Zeisler of the Inorganic Analytical Research Division, National Bureau of Standards.

J. B. Bodkin, J. C. DeVine, and N. H. Suhr of the Mineral Constitution Laboratories, The Pennsylvania State University, University Park, Pa.

S. S. Goldich of the Department of Geology, Northern Illinois University, DeKalb, Ill.

M. D. Glascock, C. C. Graham, J. R. Vogt, University of Missouri, Columbia, Mo.

The constituents given in Table 2 are *not certified* but are included for information only.

Table 2. Information Values

Constituent <sup>1</sup>	Content wt %	Constituent <sup>1</sup>	Content wt (µg/g)
C (Total Carbon) <sup>j</sup>	(0.05)	Ba <sup>f</sup>	(1140 )
CO <sub>2</sub> <sup>d</sup>	(0.01)	B <sup>g</sup>	( 25 )
F <sup>h</sup>	(0.05)	Ce <sup>f</sup>	( 62.2 )
MgO <sup>d</sup>	(0.23)	Co <sup>f</sup>	( 1.5 )
		Cr <sup>f</sup>	( 6.1 )
		Cs <sup>f</sup>	( 5.5 )
		Eu <sup>f</sup>	( 0.84 )
		Gd <sup>g</sup>	( 5.3 )
		Hf <sup>f</sup>	( 8.4 )
		Lu <sup>f</sup>	( 0.73 )
		Sb <sup>f</sup>	( 1.5 )
		Sc <sup>f</sup>	( 5.1 )
		Sm <sup>f,g</sup>	( 5.7 )
		Ta <sup>f</sup>	( 1.2 )
		Tb <sup>f</sup>	( 1.0 )
		Yb <sup>f</sup>	( 4.5 )
		Zn <sup>f</sup>	( 55 )

U.S. Department of Commerce  
 Rogers C. Merton,  
 Secretary  
 National Bureau of Standards  
 Ernest Ambler, Acting Director

# National Bureau of Standards

## Certificate of Analysis

### Standard Reference Materials 113a and 329

#### Zinc Concentrates

These Standard Reference Materials are in the form of fine powder (<15 mm) and are intended for use both in checking chemical methods of analysis and in calibration with instrumental methods of analysis.

**CAUTION:** The bottle should be kept tightly closed except when in direct use. Store in a desiccator over desiccant.

SRM No. Element	113a	329
	Percent by Weight <sup>a</sup>	
Zinc	57.3	45.5
Lead	2.80	6.06
Iron	2.08	12.94
Calcium Oxide	1.19	0.08
Magnesium Oxide	0.75	.16 <sub>5</sub>
Cadmium	.78	.14
Copper	.31	.13 <sub>2</sub>
Cobalt	(.11) <sup>b</sup>	(.009)
Nickel	(.07)	(.006)
Sulfur (Total)	30.6	(31.7)
Silicon Dioxide	(1.54)	(0.61)
Indium	---	.019
Silver	0.046 <sub>7</sub> <sup>d</sup>	.0089 <sup>d</sup>

<sup>a</sup> Based on samples dried at 105 °C for one hour. Moisture loss: for SRM 113a 0.08%; for SRM 329 0.4%.

<sup>b</sup> Figures in parenthesis are not certified but are given for information only.

<sup>c</sup> Not determined.

<sup>d</sup> Troy ounces per ton is 13.6<sub>3</sub> for SRM 113a and 2.6<sub>0</sub> for SRM 329.

**NOTE:** The total of constituents reported for SRM 113a is 97.6% and that for SRM 329 is 98.0%. The remainder is expected to consist mainly of oxygen, carbon, and water of crystallization.

**CERTIFICATION:** The value listed for a certified element is the present best estimate of the "true" value based on the results of the analytical program. The value listed is not expected to deviate from the "true" value by more than ± 1 in the last significant figure reported. For a subscript figure, the deviation is not expected to be more than ± 5. Based on the results of homogeneity testing, maximum variations within and among samples are estimated to be less than the uncertainty figures given above.

Washington, D.C. 20234  
 December 29, 1975  
 (Revision of Certificate of May 6, 1974 for  
 change in silver results)

J. Paul Cali, Chief  
 Office of Standard Reference Materials

(over)

The material for these standards was supplied by Cominco American Inc., Spokane, Wash. The material for SRM 113a was prepared at the Magmont Mines, Bixby, Mo., and that for SRM 329 at the Sullivan Mine, Trail, B.C., Canada.

Following sieving and blending operations at NBS, homogeneity testing was performed by S. D. Raspberry and J. McKay, (x-ray fluorescence analyses); by E. J. Maienthal, (polarographic analyses); and by R. K. Bell, (chemical analyses).

Selected samples representative of the lot were analyzed and no significant variability was observed when using subsamples of 0.5 g or larger. (Moisture determinations usually were made on larger samples-up to 10 g.)

Cooperative analyses for certification were performed in the analytical laboratories of Cominco, Sullivan Mine, Trail, B.C., Canada, C. J. Mitchell; Cominco American Inc., Magmont Mines, Bixby, Mo., R. J. Gibson; and St. Joe Minerals Corp., Zinc Smelting Division, Monaca, Pa., J. J. Aldrich.

Analyses were performed in the NBS Analytical Chemistry Division by R. K. Bell and E. J. Maienthal.

The overall direction and coordination of the technical measurements at NBS leading to certification were performed under the direction of O. Menis and J. I. Shultz.

The technical and support aspects involved in the preparation, certification, and issuance of these SRM's were coordinated through the Office of Standard Reference Materials by R. E. Michaelis.

U.S. Department of Commerce  
 Elliot L. Richardson,  
 Secretary

National Bureau of Standards  
 Ernest Ambler, Acting Director

# National Bureau of Standards

## Certificate of Analysis

### Standard Reference Material 330

#### Copper Ore, Mill Heads

This material is in the form of fine powder intended for use both in checking chemical methods of analysis and in calibration with instrumental methods of analysis.

This SRM is one of a series of four SRM's issued primarily for use in evaluation of the critically important "material balance" in the copper mining and metallurgical industries. The other three are: SRM 331, Copper Ore, Mill Tails; SRM 332, Copper Concentrate; and SRM 333, Molybdenum Concentrate.

**CAUTION:** The bottle should be kept tightly closed except when in direct use. Store in a desiccator over desiccant.

Constituent	Certified Value <sup>a, b</sup>	Estimated Uncertainty <sup>c</sup>
	<u>Percent by Weight</u>	
Total Copper	0.84	0.01
Molybdenum	.018	.001
	<u>PPM by Weight</u>	
Rhenium	0.30	0.06

<sup>a</sup> Based on samples dried at 105 °C for two hours. Separate samples are used for rhenium and calculated to a dry-weight basis.

<sup>b</sup> The certified value is the *best estimate* of the "true" value.

<sup>c</sup> Estimated uncertainty includes both method imprecision and material variability with samples 0.5 g (or more) for total copper, 1.0 g (or more) for molybdenum, and 2.5 g (or more) for rhenium.

The following values indicate the results of the analytical tests made at NBS and the Magma Copper Company.

Constituent / Method <sup>1</sup>	Average	Standard Deviation <sup>2</sup>	Number of Determinations
	<u>Percent by Weight</u>		
<u>Total Copper</u>			
Isotopic dilution mass spectrometry <sup>1</sup> (0.5 g samples)	0.837	0.007	8
Polarographic (0.5 g samples)	.85	.01	3
Iodometric (2.5 g samples) <sup>4</sup>	.85	.01	21
<u>Molybdenum</u>		<u>Range</u>	
Isotopic dilution mass spectrometry <sup>1</sup> (1 g samples)	.0180	0.0001	2
		<u>PPM by Weight</u>	
<u>Rhenium</u>		<u>Range</u>	
Isotopic dilution mass spectrometry <sup>1</sup> (2.5 g samples)	0.303	0.054	6

<sup>1</sup> Details of the methods used, including drying and dissolution procedures, are given in a separate publication [1].

<sup>2</sup> Of single determinations for total Cu; range given for Mo and Re.

<sup>3</sup> This method has been studied extensively and the data are considered free from systematic errors [2].

<sup>4</sup> Results from Magma Copper Company.

Washington, D. C. 20234  
 January 20, 1977  
 (Revision of Provisional  
 Certificate of 2-20-73)

J. Paul Cali, Chief  
 Office of Standard Reference Materials

(Over)



PLANNING, PREPARATION, TESTING, ANALYSIS: The material for this SRM (330) was carefully selected and provided to NBS by Magma Copper Company, San Manuel, Arizona, through the courtesy of T. L. Young.

At NBS this material was sieved and thoroughly blended, which involved several independent procedures [1].

Homogeneity testing of selected samples representative of the lot of SRM 330 was performed simultaneously with the analytical program for certification. At NBS, 0.5 g samples showed the maximum variability for total copper to be  $\pm 0.010$  percent. At Magma, 2.5 g samples showed the maximum variability to be  $\pm 0.01$  percent.

Cooperative analyses were performed at the Magma Copper Company, San Manuel, Arizona, by B. Cripe, R. L. Culder, A. B. Hall, D. A. Shah, J. T. Tadano, and M. Toelkes.

Analyses were performed in the NBS Analytical Chemistry Division by E. L. Garner, J. W. Gramlich, L. A. Machlan, E. J. Maienthal, J. R. Moody, and T. J. Murphy.

The overall direction and coordination of the technical measurements at NBS leading to certification were performed under the direction of W. R. Shields and I. L. Barnes.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. E. Michaelis.

ADDITIONAL INFORMATION ON THE COMPOSITION: Certification is made *only* for total Cu, Mo, and Re. Although NOT CERTIFIED, the following additional information on the composition may be of interest.

#### Gold and Silver

Fire assay determinations for Au and Ag were made at Magma Copper Company:

Fire assay	Gold	Silver
	PPM by Weight	
	(0.093)	*(1.51)

\*Revised 02/28/79

#### "Acid-Soluble" Copper

The total copper content includes "recoverable" sulfide copper and "nonrecoverable" oxide copper. Industrial practice is to determine "acid-soluble" copper and to relate this result to the oxide copper content. Investigation at NBS provided additional information on "acid-soluble" copper that may be useful, but is *not certified*:

Constituent/Method	Average	Range	Number of Determinations
<u>"Acid-Soluble" Copper</u> Isotopic dilution mass spectrometry (2.5 g samples)	<u>Percent by Weight</u>		9
	(0.069)	(0.063-0.081)	

#### REFERENCES

- [1] J. R. Moody, I. L. Barnes, and R. E. Michaelis, Standard Reference Materials: Copper Ore, Mill Heads - SRM 330; Copper Ore, Mill Tails - SRM 331; Copper Concentrate - SRM 332, and Molybdenum Concentrate - SRM 333; Nat. Bur. Stand. Spec. Publ. 260-xx (in press).
- [2] W. R. Shields, Editor, Nat. Bur. Stand. Tech. Note 546, (1970).

U.S. Department of Commerce  
 Elliot L. Richardson,  
 Secretary

National Bureau of Standards  
 Ernest Ambler, Acting Director

# National Bureau of Standards

## Certificate of Analysis

### Standard Reference Material 331

#### Copper Ore, Mill Tails

This material is in the form of fine powder intended for use both in checking chemical methods of analysis and in calibration with instrumental methods of analysis.

This SRM is one of a series of four SRM's issued primarily for use in evaluation of the critically important "material balance" in the copper mining and metallurgical industries. The other three are: SRM 330, Copper Ore, Mill Heads; SRM 332, Copper Concentrate; and SRM 333, Molybdenum Concentrate.

**CAUTION:** The bottle should be kept tightly closed except when in direct use. Store in a desiccator over desiccant.

Constituent	Certified Value <sup>a, b</sup>	Estimated Uncertainty <sup>c</sup>
	<u>Percent by Weight</u>	
Total Copper	0.091	0.001
Molybdenum	.0022	.0002
	<u>P P M by Weight</u>	
Rhenium	.04	.02

<sup>a</sup> Based on samples dried at 105 °C for two hours. Separate samples are used for rhenium and calculated to a dry-weight basis.

<sup>b</sup> The certified value is the best estimate of the "true" value.

<sup>c</sup> Estimated uncertainty includes both method imprecision and material variability with samples 1.0 g (or more) for total copper and molybdenum, and 2.5 g (or more) for rhenium.

The following values indicate the results of the analytical tests:

Constituent/ Method <sup>1</sup>	Average	Standard Deviation <sup>2</sup>	Number of Determinations
	<u>Percent by Weight</u>		
<u>Total Copper</u>			
Isotopic dilution mass spectrometry <sup>3</sup> (1.0 g samples)	0.0915	0.0005	15
Polarographic (2.5 g samples)	.091	.001	4
<u>Molybdenum</u>		<u>Range</u>	
Isotopic dilution mass spectrometry <sup>3</sup> (1.0 g samples)	.0022	0.0001	2
	<u>P P M by Weight</u>		
<u>Rhenium</u>		<u>Range</u>	
Isotopic dilution mass spectrometry <sup>3</sup> (2.5 g samples)	0.043	0.016	6

<sup>1</sup> Details of the methods used, including drying and dissolution procedures, are given in a separate publication [1].

<sup>2</sup> Of single determinations for total Cu, range given for Mo and Re.

<sup>3</sup> This method has been studied extensively and the data are considered free from systematic errors [2].

Washington, D. C. 20234  
 January 20, 1977  
 (Revision of Provisional  
 Certificate of 2-20-73)

J. Paul Cali, Chief  
 Office of Standard Reference Materials

(Over)

PLANNING, PREPARATION, TESTING, ANALYSIS: The material for this SRM (331) was carefully selected and provided to NBS by Magma Copper Company, San Manuel, Arizona, through the courtesy of T. L. Young.

At NBS, this material was sieved and thoroughly blended, which involved several independent procedures [1].

Homogeneity testing of selected samples representative of the lot of SRM 331 was performed simultaneously with the analytical program for certification. At NBS, the maximum variability for total copper was determined to be  $\pm 0.0008$  percent (15 determinations with 1 g samples).

Analyses were performed in the NBS Analytical Chemistry Division by E. L. Garner, J. W. Gramlich, L. A. Machlan, E. J. Maienthal, J. R. Moody, L. J. Moore, and T. J. Murphy.

The overall direction and coordination of the technical measurements at NBS leading to certification were performed under the direction of W. R. Shields and I. L. Barnes.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. E. Michaelis.

ADDITIONAL INFORMATION ON THE COMPOSITION: Certification is made *only* for total Cu, Mo, and Re. Although NOT CERTIFIED, the following additional information on the composition may be of interest.

#### Gold and Silver

Fire assay determinations for Au and Ag were made at Magma Copper Company:

Fire assay	Gold	Silver
	PPM by Weight	
	(0.034)	*(0.243)

\*Revised 02/28/79

#### "Acid-Soluble" Copper

The total copper content includes "recoverable" sulfide copper and "nonrecoverable" oxide copper. Industrial practice is to determine "acid-soluble" copper and to relate this result to the oxide copper content. Investigation at NBS provided additional information on "acid-soluble" copper that may be useful, but is *not certified*:

Constituent/Method	Average	Range	Number of Determinations
<u>"Acid-Soluble" Copper</u>	Percent by Weight		3
Isotopic dilution mass spectrometry (2.5 g samples)	(0.051)	(0.051-0.052)	

#### REFERENCES

- [1] J. R. Moody, I. L. Barnes, and R. E. Michaelis, Standard Reference Materials: Copper Ore, Mill Heads - SRM 330; Copper Ore, Mill Tails - SRM 331; Copper Concentrate - SRM 332, and Molybdenum Concentrate - SRM 333, Nat. Bur. Stand. Spec. publ. 260-xx (in press).
- [2] W. R. Shields, Editor, Nat. Bur. Stand. Tech. Note 546, (1970).

# National Bureau of Standards

## Certificate of Analysis

### Standard Reference Material 332

#### Copper Concentrate

This material is in the form of fine powder intended for use both in checking chemical methods of analysis and in calibration with instrumental methods of analysis.

This SRM is one of a series of four SRM's issued primarily for use in evaluation of the critically important "material balance" in the copper mining and metallurgical industries. The other three are: SRM 330, Copper Ore, Mill Heads; SRM 331, Copper Ore, Mill Tails; and SRM 333, Molybdenum Concentrate.

**CAUTION:** The bottle should be kept tightly closed except when in direct use. Store in a desiccator over desiccant.

Constituent	Certified Value <sup>a</sup> <sup>b</sup>	Estimated Uncertainty <sup>c</sup>
	<u>Percent by Weight</u>	
Total Copper	28.4	0.1
Molybdenum	0.64	.01
	<u>PPM by Weight</u>	
Rhenium	10.2	0.2

<sup>a</sup> Based on samples dried at 105 °C for two hours. Separate samples are used for rhenium and calculated to a dry-weight basis.

<sup>b</sup> The certified value is the *best estimate* of the "true" value.

<sup>c</sup> Estimated uncertainty includes both method imprecision and material variability with samples 0.5 g (or more) for total copper, 0.2 g (or more) for molybdenum and 2.5 g (or more) for rhenium.

The following values indicate the results of the analytical tests:

Constituent/Method <sup>1</sup>	Average	Standard Deviation <sup>2</sup>	Number of Determinations
	<u>Percent by Weight</u>		
<u>Total Copper</u>			
Isotopic dilution mass spectrometry <sup>3</sup> (0.5 g samples)	28.40	0.04	12
Electrogravimetry (2 g samples)	28.39	.02	6
<u>Molybdenum</u>		<u>Range</u>	
Isotopic dilution mass spectrometry <sup>3</sup> (0.2 g samples)	0.639	0.008	3
	<u>PPM by Weight</u>		
<u>Rhenium</u>		<u>Range</u>	
Isotopic dilution mass spectrometry <sup>3</sup> (2.5 g samples)	10.20	0.20	3

<sup>1</sup> Details of the methods used, including drying and dissolution procedures, are given in a separate publication [1].

<sup>2</sup> Of single determinations for total Cu; range given for Mo and Re.

<sup>3</sup> This method has been studied extensively and the data are considered free from systematic errors. [2].

Washington, D.C. 20234

June 26, 1977

(Revision of Provisional Certificate of 2-20-73

and draft certificates of 7-4-76 and 1-20-77)

(Over)

J. Paul Cali, Chief  
 Office of Standard Reference Materials

PLANNING, PREPARATION, TESTING, ANALYSIS: The material for this SRM (332) was carefully selected and provided to NBS by Magma Copper Company, San Manuel, Arizona, through the courtesy of T. L. Young.

At NBS, the material was sieved and thoroughly blended, which involved several independent procedures [1].

Homogeneity testing of selected samples representative of the lot of SRM 332 was performed simultaneously with the analytical program for certification. The maximum variability for total copper was determined to be  $\pm 0.06$  percent (0.5 g samples).

Analyses were performed in the NBS Analytical Chemistry Division by R. K. Bell, E. L. Garner, J. W. Gramlich, L. A. Machlan, J. R. Moody, L. J. Moore, and T. J. Murphy.

The overall direction and coordination of the technical measurements at NBS leading to certification were performed under the direction of W. R. Shields and I. L. Barnes.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. E. Michaelis.

ADDITIONAL INFORMATION ON THE COMPOSITION: Certification is made *only* for total Cu, Mo, and Re. Although NOT CERTIFIED, the following additional information on the composition may be of interest.

#### Gold and Silver

Fire assay determinations for Au and Ag were made at Magma Copper Company:

	Gold	Silver
	PPM by Weight	
	*(2.14)	*(38.7)
Fire assay		

\*Revised 02/28/79

#### REFERENCES

- [1] J. R. Moody, I. L. Barnes, and R. E. Michaelis, Standard Reference Materials: Copper Ore, Mill Heads - SRM 330; Copper Ore, Mill Tails - SRM 331; Copper Concentrate - SRM 332; and Molybdenum Concentrate - SRM 333, Nat. Bur. Stand. Spec. Publ. 260-xx (in press).
- [2] W. R. Shields, Editor, Nat. Bur. Stand. Tech. Note 546, (1970).

# National Bureau of Standards

## Certificate of Analysis

### Standard Reference Material 333

#### Molybdenum Concentrate

This material is in the form of fine powder intended for use both in checking chemical methods of analysis and in calibration with instrumental methods of analysis.

This SRM is one of a series of four SRM's issued primarily for use in evaluation of the critically important "material balance" in the copper mining and metallurgical industries. The other three are: SRM 330, Copper Ore, Mill Heads; SRM 331, Copper Ore, Mill Tails; and SRM 332, Copper Concentrate.

**CAUTION:** The bottle should be kept tightly closed except when in direct use. Store in a desiccator over desiccant.

Constituent	Certified Value <sup>a b</sup>	Estimated Uncertainty <sup>c</sup>
	Percent by Weight	
Total Copper	1.038	0.010
Molybdenum	55.3	.1
Rhenium	0.087	.001

<sup>a</sup> Based on samples run "as received." **CAUTION:** The bottle should be kept tightly closed except when in direct use.

<sup>b</sup> The certified value is the *best estimate* of the "true" value.

<sup>c</sup> Estimated uncertainty includes both method imprecision and material variability with samples 0.25 g (or more) for total copper, molybdenum, and rhenium.

The following values indicate the results of the analytical tests made at NBS and the Magma Copper Company.

Constituent/Method <sup>1</sup>	Average	Standard Deviation <sup>2</sup>	Number of Determinations
	Percent by Weight		
<u>Total Copper</u>			
Isotopic dilution mass spectrometry <sup>3</sup> (0.25 g samples)	1.038	0.007	11
Polarographic (1.0 g samples)	1.034	.011	6
Atomic absorption spectrometry <sup>4</sup> (1.0 g samples)	1.033	.002	3
<u>Molybdenum</u>			
Isotopic dilution mass spectrometry <sup>3</sup> (0.25 g samples)	55.31	.02	10
$\alpha$ -Benzoinoxime gravimetric <sup>5</sup> (0.25 g samples)	55.43	.04	10
<u>Rhenium</u>			
Isotopic dilution mass spectrometry <sup>3</sup> (0.25 g samples)	0.0869	0.0003	17
Thermal neutron activation analysis <sup>6</sup> (0.15 g samples)	.085	.002	9

<sup>1</sup> Details of the methods used are given in a separate publication [1].

<sup>2</sup> Of single determinations.

<sup>3</sup> This method has been studied extensively and the data are considered free from systematic errors [2].

<sup>4</sup> Results from Magma Copper Company.

<sup>5</sup> Average of two independent analysts at NBS.

<sup>6</sup> Determinations made relative to an industrial molybdenum concentrate reference material containing 0.1140 wt. % of Re, (as determined by a number of industrial laboratories and by isotopic dilution mass spectrometry at NBS.)

Washington, D. C. 20234  
 January 20, 1977  
 (Revision of Provisional  
 Certificate of 2-20-73)

J. Paul Cali, Chief  
 Office of Standard Reference Materials

(Over)

NOTE: Recommendation is made that this material be analyzed in the "as received" condition. SRM 333 contains a small quantity (about 1%) of fuel oil introduced in the flotation process. Investigations at NBS indicate that the analytical results made on the "as received" samples may be calculated to the dry-weight basis by use of an acetone stripping procedure (includes fuel oil and moisture)[1]. (For information only, a calculated MoS<sub>2</sub> content thus would be 93.2±0.2%.)

ADDITIONAL INFORMATION ON THE COMPOSITION: Although certification is made only for total Cu, Mo, and Re, recommended values for Au and Ag are given below:

	Au	Ag
	ppm by weight	
Fire Assay, Magma (14.58 g samples)	8.9	25.0

Neutron activation values at NBS on 0.2 g portions (final blended material) gave values of 12.5 ppm Au and 22.6 ppm Ag. Definite evidence of inhomogeneity was observed for Au on these 0.2 g portions. Recommendation is made that sample sizes of 2.5 g (or more) be used to ensure homogeneity (<5% relative), regardless of the method used.

PLANNING, PREPARATION, TESTING, ANALYSIS: The material for this SRM was carefully selected and provided to NBS by Magma Copper Company, San Manuel, Arizona, through the courtesy of T. L. Young.

At NBS, highly specialized blending and mixing procedures were employed to obtain satisfactory homogeneity. Extensive chemical analyses performed by Magma both for total copper and for molybdenum sulfide demonstrated that homogeneity was not achieved until the total blending and mixing procedures were performed three separate times [1].

Final homogeneity testing was performed at NBS simultaneously with the analytical program for certification. The maximum variability of the accepted lot was determined to be ±0.010 percent for total copper, ±0.0010 percent for rhenium; and ±0.06 percent for molybdenum (all using 0.25 g samples).

Cooperative analyses were performed at the Magma Copper Company, San Manuel, Arizona, by Y. Arias, B. Cripe, R. L. Culver, A. B. Hall, D. A. Shah, J. T. Tadano, and M. Toelkes.

Analyses were performed in the NBS Analytical Chemistry Division by R. K. Bell, E. L. Garner, T. E. Gills, J. W. Gramlich, P. D. LaFleur, L. A. Machlan, E. J. Maienthal, J. R. Moody, L. J. Moore, and T. J. Murphy.

The overall direction and coordination of the technical measurements at NBS leading to certification were performed under the direction of W. R. Shields and I. L. Barnes.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. E. Michaelis.

## REFERENCES

- [1] J. R. Moody, I. L. Barnes, and R. E. Michaelis, Standard Reference Materials: Copper Ore, Mill Heads - SRM 330; Copper Ore, Mill Tails - SRM 331; Copper Concentrate - SRM 332, and Molybdenum Concentrate - SRM 333, Nat. Bur. Stand. Spec. Publ. 260-xx (in press).
- [2] W. R. Shields, Editor, Nat. Bur. Stand. Tech. Note 546, (1970).



# National Bureau of Standards

## Certificate

### Standard Reference Material 688

#### Basalt Rock

This Standard Reference Material (SRM) is intended for use in evaluating the accuracy of analytical methods and instruments used in the analysis of geological type materials. SRM 688 is a finely powdered basalt rock that was obtained from a Cenozoic basalt flow near Jackpot, Nevada.

#### Certified Values of Constituents

The concentrations of the constituents were determined by methods that are widely used in the field of geological analysis and have a demonstrated accuracy. The values given are "certified" values, i.e., those values that were determined by either a definitive method, reference method, or by two or more independent methods, and "information" values that were determined by single or non-reference methods. The certified values are given in Table 1.

Table 1 Certified Values of Constituents

Constituent <sup>1</sup>	Content <sup>2</sup> wt (%)	Constituent <sup>1</sup>	Content <sup>2</sup> wt (μg/g)
Al <sub>2</sub> O <sub>3</sub> <sup>c,g</sup>	17.36 ± 0.09	Cr <sup>b,e</sup>	332 ± 9
FeO <sup>g</sup>	7.64 ± 0.03	Rb <sup>d</sup>	1.91 ± 0.01
Fe <sub>2</sub> O <sub>3</sub> <sup>c,g</sup>	10.35 ± 0.04	Sr <sup>d</sup>	169.2 ± 0.7
(Total Fe as Fe <sub>2</sub> O <sub>3</sub> )		Th <sup>d</sup>	0.33 ± 0.02
K <sub>2</sub> O <sup>b,d</sup>	0.187 ± 0.008	Pb <sup>d</sup>	3.3 ± 0.2
MnO <sup>a,b,c</sup>	0.167 ± 0.002		
Na <sub>2</sub> O <sup>b,c,e</sup>	2.15 ± 0.03		
P <sub>2</sub> O <sub>5</sub> <sup>a,c</sup>	0.134 ± 0.003		
SiO <sub>2</sub> <sup>c</sup>	48.4 ± 0.1		
TiO <sub>2</sub> <sup>a,b</sup>	1.17 ± 0.01		

1. Methods of Analysis

<sup>a</sup>Colorimetry

<sup>b</sup>Emission spectrometry

<sup>c</sup>Gravimetry

<sup>d</sup>Isotope dilution mass spectrometry

<sup>e</sup>Neutron activation analysis

<sup>f</sup>Specific ion electrode potentiometry

<sup>g</sup>Titrimetry

2. The estimated uncertainties of the certified values are based on judgment and represent an evaluation of the combined effects of method imprecision, possible systematic errors among methods and material variability of 250 mg or more. (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determination of most constituents.)

The overall direction and coordination of the technical measurements leading to certification were performed in the Inorganic Analytical Research Division, E. L. Garner, Chief.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. E. Gills.

Washington, D.C. 20234  
 August 18, 1981

George A. Uriano, Chief  
 Office of Standard Reference Materials

(over)



## SUPPLEMENTAL INFORMATION

### Preparation

The material was processed by the Colorado School of Mines, Golden, Colorado. Approximately 600 pounds of basalt rock were crushed, ground, and sieved to <200 mesh. The material was mixed in a cone blender to ensure homogeneity. The inhomogeneity was tested by taking random samples and analyzing for both major and minor constituents and was found to be  $\leq 2\%$  relative.

### Analysis

SRM 688 may pick up additional water on exposure to the atmosphere. Thus, exposure time should be kept to a minimum. Before analysis, it is recommended that the material be dried at 105 °C for 24 hours. Typical weight loss upon drying is approximately 0.2 percent.

The analysts and laboratories cooperating in the analytical program for certification were:

I. L. Barnes, M. J. Blackman, E. L. Garner, J. W. Gramlich, L. A. Machlan, L. J. Moore, and R. Zeisler of the Inorganic Analytical Research Division, National Bureau of Standards.

J. B. Bodkin, J. C. DeVine, and N. H. Suhr of the Mineral Constitution Laboratories, The Pennsylvania State University, University Park, Pa.

S. S. Goldich of the Department of Geology, Northern Illinois University, Dekalb, Ill.

The constituents given in Table 2 are *not certified*, but are included for information only.

Table 2 Information Values

<u>Constituent</u> <sup>1</sup>	<u>Content wt %</u>	<u>Constituent</u> <sup>1</sup>	<u>Content wt (μg/g)</u>
CaO <sup>c</sup>	(12.17)	Ce <sup>e</sup>	(13.3)
CO <sub>2</sub> <sup>f</sup>	(0.05)	Co <sup>e</sup>	(49.7)
F <sup>f</sup>	(0.02)	Eu <sup>e</sup>	( 1.07)
MgO <sup>c</sup>	(8.4 )	Hf <sup>e</sup>	( 1.6)
		Lu <sup>e</sup>	( 0.34)
		Sc <sup>e</sup>	(38.1)
		Ba <sup>b</sup>	(200)
		V <sup>b</sup>	(250)
		Cu <sup>b</sup>	(96)
		Ni <sup>b</sup>	(150)
		Sm <sup>e</sup>	(2.79)
		Tb <sup>e</sup>	(0.448)
		U <sup>d</sup>	(0.37)
		Yb <sup>e</sup>	(2.09)
		Zn <sup>e</sup>	(58.0)

# National Bureau of Standards

## Certificate of Analysis

### Standard Reference Material 690

### Iron Ore Concentrate (Canada)

(In Cooperation with the American Society for Testing and Materials)

This material is in the form of powder ( $<0.1$  mm) for use in checking chemical methods of analysis and in calibration with instrumental methods of analysis.

(Results based on samples dried for one hour at  $105^{\circ}\text{C}$ .)

Constituent	Total Fe	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	P	S	TiO <sub>2</sub>	MnO	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O
Certified <sup>1</sup> Value (wt. %)	66.85	3.71	0.18	0.011	0.003	0.022	0.23	0.20	0.18	0.003	0.0030
Estimated <sup>2</sup> Uncertainty	0.07	0.02	0.01	0.002	0.001	0.002	0.01	0.01	0.01	0.001	0.0005
Method <sup>3</sup>											
Labs	1 SrCl <sub>2</sub> K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub>	2 HClO <sub>4</sub> Dehydration	3 Atomic Absorption	4 Photometric	5 Combustion- Titration	6 Photometric	7 Atomic Absorption	8 Atomic Absorption	9 Atomic Absorption	10 Atomic Absorption	11 Atomic Absorption
A	<sup>a</sup> 66.91	<sup>b</sup> 3.70	0.17	<sup>c</sup> 0.009	$<0.005$	<sup>d</sup> 0.021	0.24	0.20	0.17	0.0026	0.0030
B	66.88	3.76	.17	<sup>e</sup> .013	.003	<sup>f</sup> .026	.23	.20	.19	.004	.003
C	<sup>g</sup> 66.82	3.70	.19	.011	.006	<sup>h</sup> .024	<sup>i</sup> .23	<sup>j</sup> .19	<sup>k</sup> .19	.0023	<sup>l</sup> .0034
D	66.85	3.73	<sup>m</sup> .18	-	-	<sup>n</sup> .021	.23	.21	.18	<sup>o</sup> .0030	<sup>p</sup> .0029
E	66.83	<sup>q</sup> 3.69 <sup>r</sup> 3.73	<sup>s</sup> .20	.009	.002	<sup>t</sup> .022	.24	.21	.18	.002	.003

- The certified value listed for a constituent is the *present best estimate* of the "true" value based on results of the cooperative analytical program for certification.
- The estimated uncertainty is based on judgment and represents an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material variability for samples of 0.5 g or more. (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determinations.)
- A detailed description of many of the methods of analysis employed in the certification program for this SRM may be found in Part 12, Chemical Analysis of Metals and Metal Bearing Ores, Annual Book of ASTM Standards.

a H<sub>2</sub>S reduction  
 b Sample fused in Na<sub>2</sub>CO<sub>3</sub>  
 c Alkali-molybdate method  
 d H<sub>2</sub>O<sub>2</sub> photometric  
 e Atomic absorption  
 f Silver reductor

g Chromotropic acid photometric  
 h Photometric method  
 i Spectroscopic method  
 j Flame emission  
 k Chromazurol S photometric

Washington, D.C. 20234  
 October 24, 1978

J. Paul Cali, Chief  
 Office of Standard Reference Materials

(over)

#### PLANNING, PREPARATION, TESTING, ANALYSIS:

The iron ore powder concentrate material for this SRM was prepared in final powder form, minus 74  $\mu\text{m}$  (200 mesh), by the Iron Ore Company of Canada, Labrador City, Newfoundland, Canada, through the courtesy of L. Rompré.

At NBS, the material was resieved and thoroughly blended.

Homogeneity testing of selected samples representative of the final lot was performed at NBS by R. K. Bell, Assistant Research Associate, ASTM-NBS Research Associate Program. The results for iron indicate that the material variability (0.5 g samples) is  $\leq$  the method imprecision.

Chemical analyses for certification were performed in the following laboratories:

Bethlehem Steel Corporation, Homer Research Laboratories, Bethlehem, Pa., D. A. Flinchbaugh.  
Inland Steel Company, Indiana Harbor Works, East Chicago, Indiana, J. E. Joyce.  
Ledoux and Company, Teaneck, New Jersey, S. Kallman and C. L. Maul.  
National Bureau of Standards, Center for Analytical Chemistry, Washington, D.C., T. C. Rains, T. J. Brady,  
J. D. Messman, and T. A. Rush and by R. K. Bell, ASTM Assistant Research Associate.  
STELCO, The Steel Company of Canada, Ltd., Hilton Works, Hamilton, Ontario, Canada, O. P. Bhargava.

The overall direction and coordination of the technical measurements leading to certification were performed jointly by R. E. Michaelis, Office of Standard Reference Materials and by J. I. Shultz, Research Associate, ASTM-NBS Research Associate Program.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by W. P. Reed.

# National Bureau of Standards

## Certificate of Analysis

### Standard Reference Material 691

#### Reduced Iron Oxide

(In cooperation with the American Society for Testing and Materials)

This Standard Reference Material (SRM) is intended for use in evaluating chemical methods and in calibrations associated with optical emission and x-ray spectrometric methods of analysis. SRM 691 is a finely powdered material (-200 mesh) and must be kept in a tightly sealed bottle when not in use. It is recommended that the material be stored in a desiccator over a suitable desiccant when not in use. Long term (>3 years) stability of this SRM has not been rigorously established. NBS will continue to monitor this material and any substantive changes will be reported to users.

The certified values given below in Table 1 are based on samples of at least 0.5 g, the minimum amount to be used for analysis. Non-certified values which are given for information only, are listed in Table 2.

Table 1

Constituent	Certified Value, <sup>1</sup> % by wt.	Estimated Uncertainty <sup>2</sup>	Constituent	Certified Value, <sup>1</sup> % by wt.	Estimated Uncertainty <sup>2</sup>
Iron (Total)	90.8	± 0.5	Copper	0.032	± 0.003
Iron (Metallic) <sup>3</sup>	84.6	.6	Cobalt	.030	.007
SiO <sub>2</sub>	3.7	.2	Phosphorus	.006	.001
Al <sub>2</sub> O <sub>3</sub>	1.22	.07	Sulfur	.008	.001
TiO <sub>2</sub>	0.27	.04	Carbon (Total)	.12	.03
CaO	.63	.03			
MnO	.043	.002			
MgO	.52	.02			
Na <sub>2</sub> O	.186	.002			

1. The certified value listed for a constituent is the present best estimate of the "true" value based on the results of the cooperative program for certification.

2. The estimated uncertainty listed for a constituent is based on judgment and represents an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material variability for samples 0.5 g or more. (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determination of most constituents.)

3. The metallic iron was determined by the ISO (Bromine-methanol) method.

The overall coordination of the technical measurements leading to certification was performed under the direction of J.I. Shultz, Research Associate, ASTM-NBS Research Associate Program.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T.E. Gills.

Washington, D.C. 20234  
April 12, 1982

George A. Uriano, Chief  
Office of Standard Reference Materials

(over)

# PLANNING, PREPARATION, TESTING, ANALYSIS:

The material for this SRM was provided by Allis-Chalmers, Reduction Systems Division, Milwaukee, Wis., courtesy of L.J. Wrangell. It was processed (crushed, ground, sieved, and mixed) at the Colorado School of Mines Research Institute, Golden, Colorado, under a contract to the National Bureau of Standards. The final product (-200 mesh) was blended at NBS.

Homogeneity testing of selected samples representative of the final lot was performed at Allis-Chalmers, L.J. Wrangell; at Ledoux & Co., Teaneck, New Jersey, by S. Kallmann; and at NBS by E.R. Deardorff.

Stability tests conducted over a seven-month period at NBS, during which samples were exposed to relative humidities of 75 and 90 percent at room temperature, indicated sufficient stability of the material for use as an SRM.

This material was packaged in a dry nitrogen atmosphere to prevent oxidation. If signs of oxidation are detected, please transmit this information to NBS for documentation into the monitoring program.

Cooperative analyses for certification were performed in the following laboratories:

Andrew S. McCreath & Son, Inc., Harrisburg, Pa.; F.A. Pennington, Jr., R.F. Eakin, G.L. Dobbs, J.C. Forney, and L.W. Richards.

Inland Steel Company, Indiana Harbor Works, East Chicago, Indiana; J.E. Joyce.

Institut de Recherches de la Siderurgie, Maizières-les-Metz, France; G. Jecko.

Ledoux and Company, Teaneck, New Jersey; S. Kallmann.

National Bureau of Standards, Inorganic Analytical Research Division, C.G. Blundell, T.A. Butler, E.R. Deardorff, M.S. Epstein, R.M. Lindstrom, T.C. Rains, M. Sadjadi, and R.M. Stone.

United States Steel Corp., Research Laboratory, Monroeville, Pa.; J.D. Selvaggio, D.S. Shafferman, A.W. Fioravanti, D.G. Cunningham, K.G. Mikos, R.C. Cline, and H.S. Karp.

The values shown below are *not certified* since they are not based on the results of at least two independent laboratories or methods. These values are included for information only.

Table 2

Element	Content Wt. Percent	Element	Content μg/g
Cr	(0.03)	As	(14)
Ni	(.3)	Zn	(40)
K	(.06)	Pb	( $\leq 20$ )
		Cd	( $\leq 5$ )
		Mo	( $\leq 20$ )
		Sn	( $\leq 10$ )
		N	(50)
		V	(135)

# National Bureau of Standards

## Certificate of Analysis

### Standard Reference Material 692

#### Iron Ore (Labrador)

(In Cooperation with the American Society for Testing and Materials)

This material is in the form of powder (<0.1mm) for use in checking chemical methods of analysis and in calibration with instrumental methods of analysis.

(Results based on samples dried for one hour at 105 °C.)

Constituent	Total Fe	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	P	S	TiO <sub>2</sub>	MnO	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O
Certified <sup>1</sup> Value (wt. %)	59.58	10.14	1.41	0.039	0.005	0.045	0.46	0.023	0.035	0.008	0.039
Estimated <sup>2</sup> Uncertainty	0.06	0.05	0.04	0.002	0.001	0.005	0.01	0.003	0.004	0.002	0.003
Method <sup>3</sup>	Method <sup>3</sup>	Method <sup>3</sup>	Method <sup>3</sup>	Method <sup>3</sup>	Method <sup>3</sup>	Method <sup>3</sup>	Method <sup>3</sup>	Method <sup>3</sup>	Method <sup>3</sup>	Method <sup>3</sup>	Method <sup>3</sup>
Labs	SiO <sub>2</sub> = K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub>	HClO <sub>4</sub> Dehydration	Atomic Absorption	Photometric	Combustion-Titration	Photometric	Atomic Absorption	Atomic Absorption	Atomic Absorption	Atomic Absorption	Atomic Absorption
A	<sup>a</sup> 59.55	<sup>b</sup> 10.09	1.40	<sup>c</sup> 0.039	<0.005	<sup>d</sup> 0.041	0.47	0.022	0.034	0.008	0.040
B	59.58	10.26	1.48	<sup>e</sup> 0.035	.004	<sup>e</sup> .05	.45	.02	.04	.005	.036
C	<sup>f</sup> 59.63 <sup>f</sup> 59.62	10.10	1.42	.040	.007	<sup>g</sup> .048	<sup>h</sup> .47	<sup>i</sup> .025	<sup>i</sup> .028	<sup>i</sup> .008	<sup>j</sup> .040 <sup>j</sup> .039
D	59.60	10.16	1.40	-	-	<sup>j</sup> .043	.47	<sup>j</sup> .021	.033	<sup>j</sup> .010 <sup>j</sup> .009	<sup>j</sup> .041
E	59.50	<sup>k</sup> 10.12 <sup>k</sup> 10.05	1.41	.039	.004	<sup>g</sup> .050	.46	.01	.038	.008	.041
F	59.58	10.18	<sup>l</sup> 1.37 <sup>l</sup> 1.46	.040	.005	<sup>g</sup> .043	<sup>h</sup> .46	.026	.035	.008	.035

- The certified value listed for a constituent is the *present best estimate* of the "true" value based on results of the cooperative analytical program for certification.
- The estimated uncertainty is based on judgment and represents an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material variability for samples of 0.5 g or more. (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determinations.)
- A detailed description of many of the methods of analysis employed in the certification program for this SRM may be found in Part 12, Chemical Analysis of Metals and Metal Bearing Ores, Annual Book of ASTM Standards.

- a H<sub>2</sub>S reduction  
b Sample fused in Na<sub>2</sub>CO<sub>3</sub>  
c Alkali-molybdate method  
d H<sub>2</sub>O<sub>2</sub> photometric  
e Atomic absorption  
f Silver reductor

- g Chromotropic acid photometric  
h Photometric method  
i Spectroscopic method  
j Flame emission  
k Chromazurol S photometric  
l Mercury cathode-NH<sub>4</sub>OH-Cupferron-ALPO<sub>4</sub>

Washington, D.C. 20234  
October 24, 1978

J. Paul Cali, Chief  
Office of Standard Reference Materials

(over)

#### PLANNING, PREPARATION, TESTING, ANALYSIS:

The iron ore material for this SRM was prepared in final powder form, minus 74  $\mu\text{m}$  (200 mesh), by the Bethlehem Steel Corporation, Bethlehem, Pa. through the courtesy of J. M. Karpinski.

At NBS, the material was resieved and thoroughly blended.

Homogeneity testing of selected samples representative of the final lot was performed at NBS by R. K. Bell, Assistant Research Associate, ASTM-NBS Research Associate Program. The results for iron indicate that the material variability (0.5 g samples) is  $\leq$  the method imprecision.

Chemical analyses for certification were performed in the following laboratories:

Bethlehem Steel Corporation, Homer Research Laboratories, Bethlehem, Pa., D. A. Flinchbaugh.  
Inland Steel Company, Indiana Harbor Works, East Chicago, Indiana, J. E. Joyce.  
Ledoux and Company, Teaneck, New Jersey, S. Kallman and C. L. Maul.  
National Bureau of Standards, Center for Analytical Chemistry, Washington, D.C., T. C. Rains, T. J. Brady, J. D. Messman, and T. A. Rush, and by R. K. Bell, ASTM Assistant Research Associate.  
STELCO, The Steel Company of Canada, Ltd., Hilton Works, Hamilton, Ontario, Canada, O. P. Bhargava.  
United States Steel Corporation, Research Laboratory, Monroeville, Pa., L. M. Melnick, J. D. Selvaggio, R. W. Cline, D. G. Cunningham, A. V. Fioravanti, J. R. Lucas II, C. W. Ponsonby, L. E. Povirk, D. Shafferman and R. J. Wargo.

The overall direction and coordination of the technical measurements leading to certification were performed jointly by R. E. Michaelis, Office of Standard Reference Materials, and by J. I. Shultz, Research Associate, ASTM-NBS Research Associate Program.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by W. P. Reed.

# National Bureau of Standards Certificate of Analysis Standard Reference Material 693 Iron Ore (Nimba)

(In Cooperation with the American Society for Testing and Materials)

This material is in the form of powder (<0.1mm) for use in checking chemical methods of analysis and in calibration with instrumental methods of analysis.

(Results based on samples dried for one hour at 105 °C.)

Constituent	Total Fe	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	P	S	TiO <sub>2</sub>	MnO	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O
Certified <sup>1</sup> Value (wt. %)	65.11	3.87	1.02	0.056	0.005	0.035	0.091	0.016	0.013	0.0028	0.0028
Estimated <sup>2</sup> Uncertainty	0.07	0.02	0.04	0.001	0.001	0.003	0.004	0.004	0.002	0.0005	0.0006
Method <sup>3</sup>	SnCl <sub>2</sub> + K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub>	HClO <sub>4</sub> Precipitation	Atomic Absorption	Photometric	Combustion- Titration	Photometric	Atomic Absorption	Atomic Absorption	Atomic Absorption	Atomic Absorption	Atomic Absorption
Labs											
A	<sup>a</sup> 65.11	<sup>b</sup> 3.87	<sup>c</sup> 1.04	<sup>e</sup> 0.056	<sup>d</sup> <0.005	<sup>d</sup> 0.034	<sup>e</sup> 0.094	<sup>f</sup> 0.016	<sup>f</sup> 0.013	<sup>g</sup> 0.0029	<sup>g</sup> 0.003
B	<sup>e</sup> 65.13 <sup>e</sup> 65.14	<sup>h</sup> 3.83	<sup>h</sup> 1.11	<sup>h</sup> .055	<sup>h</sup> .005	<sup>h</sup> .036	<sup>h</sup> .095	<sup>h</sup> .020	<sup>h</sup> .011	<sup>h</sup> .0023	<sup>h</sup> .003
C	<sup>i</sup> 65.10	<sup>i</sup> 3.89	<sup>i</sup> 1.06	<sup>i</sup> -	<sup>i</sup> -	<sup>i</sup> .032	<sup>i</sup> .087	<sup>i</sup> .015	<sup>i</sup> .012	<sup>i</sup> .0030 <sup>i</sup> .0033	<sup>i</sup> .003
D	<sup>j</sup> 65.11	<sup>j</sup> 3.86 <sup>j</sup> 3.88	<sup>j</sup> 0.98	<sup>j</sup> .056	<sup>j</sup> .005	<sup>j</sup> .038	<sup>j</sup> .090	<sup>j</sup> .01	<sup>j</sup> .015	<sup>j</sup> .003	<sup>j</sup> .003
E	<sup>k</sup> 65.09	<sup>k</sup> 3.87 <sup>k</sup> 1.06	<sup>k</sup> 0.98 <sup>k</sup> 1.06	<sup>k</sup> .056	<sup>k</sup> .007	<sup>k</sup> .033	<sup>k</sup> .091	<sup>k</sup> .018	<sup>k</sup> .015	<sup>k</sup> .002	<sup>k</sup> .002

1. The certified value listed for a constituent is the *present best estimate* of the "true" value based on results of the cooperative analytical program for certification.

2. The estimated uncertainty is based on judgment and represents an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material variability for samples of 0.5 g or more. (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determinations.)

3. A detailed description of many of the methods of analysis employed in the certification program for this SRM may be found in Part 12, Chemical Analysis of Metals and Metal Bearing Ores, Annual Book of ASTM Standards.

a H<sub>2</sub>S reduction

b Sample fused in Na<sub>2</sub>CO<sub>3</sub>

c Alkali-molybdate method

d H<sub>2</sub>O photometric

e Silver reductor

f Chromotropic acid photometric

g Photometric method

h Spectroscopic method

i Flame emission

j Chromazurol S photometric

k Mercury cathode-NH<sub>4</sub>OH-Cupferron-AlPO<sub>4</sub>

Washington, D.C. 20234  
 October 24, 1978

J. Paul Cali, Chief  
 Office of Standard Reference Materials

(over)



#### PLANNING, PREPARATION, TESTING, ANALYSIS:

The iron ore material for this SRM was prepared in final powder form, minus 74  $\mu\text{m}$  (200 mesh), by the Bethlehem Steel Corporation, Bethlehem, Pa. through the courtesy of J. M. Karpinski.

At NBS, the material was resieved and thoroughly blended.

Homogeneity testing of selected samples representative of the final lot was performed at NBS by R. K. Bell, Assistant Research Associate, ASTM-NBS Research Associate Program. The results for iron indicate that the material variability (0.5 g samples) is  $\leq$  the method imprecision.

Chemical analyses for certification were performed in the following laboratories:

Bethlehem Steel Corporation, Homer Research Laboratories, Bethlehem, Pa., D. A. Flinchbaugh.

Ledoux and Company, Teaneck, New Jersey, S. Kallman and C. L. Maul.

National Bureau of Standards, Center for Analytical Chemistry, Washington, D.C., T. C. Rains, T. J. Brady, J. D. Messman, and T. A. Rush, and by R. K. Bell, ASTM Assistant Research Associate.

STELCO, The Steel Company of Canada, Ltd., Hilton Works, Hamilton, Ontario, Canada, O. P. Bhargava. United States Steel Corporation, Research Laboratory, Monroeville, Pa., L. M. Melnick, J. D. Selvaggio, R. W. Cline, D. G. Cunningham, A. V. Fioravanti, J. R. Lucas II, C. W. Ponsonby, L. E. Povirk, D. Shafferman and R. J. Wargo.

The overall direction and coordination of the technical measurements leading to certification were performed jointly by R. E. Michaelis, Office of Standard Reference Materials and by J. I. Shultz, Research Associate, ASTM-NBS Research Associate Program.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by W. P. Reed.

U.S. Department of Commerce  
Juanita M. Kreps  
Secretary

National Bureau of Standards  
Ernest Ambler, Director

# National Bureau of Standards

## Certificate of Analysis

### Standard Reference Material 696

#### Bauxite (Surinam)

(In Cooperation with the American Society for Testing and Materials)

(All analyses are based on samples dried 2 hours at 140 °C)

This material is in the form of fine powder (<0.08 mm) for use in checking chemical and instrumental methods of analyses.

Constituent	Certified Value <sup>1</sup> Percent, by weight	Estimated Uncertainty <sup>2</sup>
Al <sub>2</sub> O <sub>3</sub>	54.5	0.3
Fe <sub>2</sub> O <sub>3</sub>	8.70	.10
SiO <sub>2</sub>	3.79	.10
TiO <sub>2</sub>	2.64	.05
ZrO <sub>2</sub>	0.14	.02
P <sub>2</sub> O <sub>5</sub>	.050	.006
V <sub>2</sub> O <sub>5</sub>	.072	.006
Cr <sub>2</sub> O <sub>3</sub>	.047	.003
CaO	.018	.002
MgO	.012	.003
MnO	.004	.001
ZnO	.0014	.0007
K <sub>2</sub> O	.009	.003
SO <sub>3</sub>	.21	.03
Loss on Ignition <sup>3</sup>	29.9	.2

<sup>1</sup>The certified value listed for a constituent is the *present best estimate* of the "true" value.

<sup>2</sup>The estimated uncertainty listed for a constituent is based on judgment and represents an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material variability for samples 1.0 g or more. (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determination of most constituents.)

<sup>3</sup>Determined by igniting to constant weight at 1050 °C.

Washington, D.C. 20234  
August 24, 1979

George A. Uriano, Chief  
Office of Standard Reference Materials

(over)

#### ADDITIONAL INFORMATION ON THE COMPOSITION

Elements other than those certified may be present in this material as indicated below. These are not certified but are given as additional information on the composition.

<u>Constituent</u>	<u>Concentration, Percent by weight</u>	<u>Constituent</u>	<u>Concentration, Percent by weight</u>
BaO	(0.004)	Co	(0.00009)
Na <sub>2</sub> O	(0.007)	Hf	(0.0032)
Ce	(0.0041)	Sc	(0.0008)

The mineralogical composition of SRM 696 was determined by x-ray diffraction studies at the Geological Survey, U.S. Department of the Interior, Reston, Va., (J.W. Hosterman) to be 5% kaolinite, 80% gibbsite, 10% pyrite, and 5% anatase. These results are semiquantitative (to the nearest 5%).

#### PLANNING, PREPARATION, TESTING, ANALYSIS:

The material for this SRM was mined in Surinam, South America, and was provided by the Aluminum Company of America, Alcoa Technical Center, Pittsburgh, Pa., through the courtesy of H. B. Hartman. It was processed (crushed, ground, sieved, and mixed) at the Colorado School of Mines Research Institute under a contract with the National Bureau of Standards.

Homogeneity testing was performed at NBS by J.S. Maples and T.E. Gills.

Cooperative analyses for certification were performed in the following laboratories:

Aluminum Company of America, Alcoa Center, Pa., R. C. Obbink.

Aluminum Company of Canada, Ltd., Arvida Research Center, Arvida, Quebec, Canada, L. Girolami.

Andrew S. McCreath & Son, Inc., Harrisburg, Pa., F. A. Pennington, Jr., R. F. Eakin, and S. L. Miller.

General Refractories Co., U.S. Refractories Division, Research Center, Baltimore, Md., S. Banerjee.

Geological Survey, U.S. Department of the Interior, Reston, Va., H. J. Rose, Jr., and J. W. Hosterman.

Kaiser Aluminum and Chemical Corp., Center for Technology, Pleasanton, Calif., H. J. Seim, A. E. McLaughlin, D. F. G. Marten, A. Kermaninejad, R. C. Kinne, J. R. Skarset, J. Boruk, and U. Vogel.

National Bureau of Standards, Washington, D.C., R. K. Bell, ASTM-NBS Assistant Research Associate.

National-Southwire Aluminum Co., Hawesville, Ky., N. Robinson and E. Gotzy.

Ormet Corp., Burnside, La., W. L. Brown and A. D. Lafleur.

Reynolds Aluminum Co., Alumina Research Division, Bauxite, Ark., J. B. Ezell, Jr.

University of Kentucky, Institute for Mining and Minerals Research, Center for Energy Research Laboratory, Lexington, Ky., T. V. Rebagay.

The overall coordination of the technical measurements leading to certification were performed under the direction of J. I. Shultz, Research Associate, ASTM-NBS Research Associate Program.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. E. Michaelis and R. Alvarez.

## NBS Standard Reference Materials

## BAUXITE SERIES

September 4, 1979

R. E. Michaelis and R. Alvarez, NBS Office of Standard Reference Materials  
and

J. I. Shultz, ASTM Research Associate

The following table gives the values for four bauxite SRM's that are available in the form of fine powder (<0.08 mm) for use in chemical and instrumental methods of analysis. They are being issued as a culmination of a major Industry-ASTM-NBS cooperative program.

SRM No. Designation	69b Arkansas	696 Surinam	697 Dominican	698 Jamaican
<u>Constituent</u>	<u>Percent by Weight</u>			
Al <sub>2</sub> O <sub>3</sub>	48.8	54.5	45.8	48.2
Fe <sub>2</sub> O <sub>3</sub>	7.14	8.70	20.0	19.6
SiO <sub>2</sub>	13.43	3.79	6.81	0.69
TiO <sub>2</sub>	1.90	2.64	2.52	2.38
ZrO <sub>2</sub>	0.29	0.14	0.065	0.061
P <sub>2</sub> O <sub>5</sub>	0.118	0.050	0.97	0.37
V <sub>2</sub> O <sub>5</sub>	0.028	0.072	0.063	0.064
Cr <sub>2</sub> O <sub>3</sub>	0.011	0.047	0.100	0.080
CaO	0.13	0.018	0.71	0.62
MgO	0.085	0.012	0.18	0.058
MnO	0.110	0.004	0.41	0.38
ZnO	0.0035	0.0014	0.037	0.029
BaO	(0.008) <sup>a</sup>	(0.004)	(0.015)	(0.008)
Na <sub>2</sub> O	(0.025)	(0.007)	(0.036)	(0.015)
K <sub>2</sub> O	0.068	0.009	0.062	0.010
SO <sub>3</sub>	0.63	0.21	0.13	0.22
Loss on Ign.	27.2	29.9	22.1	27.3
Ce	(0.024)	(0.0041)	(0.069)	(0.030)
Co	(0.0001)	(0.00009)	(0.0013)	(0.0045)
Hf	(0.0063)	(0.0032)	(0.0014)	(0.0015)
Sc	(0.0008)	(0.0008)	(0.0058)	(0.0051)
Total	(100.0)	(100.1)	(100.1)	(100.1)

<sup>a</sup>Values in parenthesis are *not certified*.

The value listed for a certified constituent is the *present best estimate* of the "true" value based on the results of the analytical program for certification (10-12 laboratories). The individual certificates of analysis list the "estimated uncertainties" associated with the certified values (also listed is a semiquantitative mineralogical composition ( $\pm 5\%$ ) as determined by x-ray diffraction studies at the U.S. Geological Survey).

Inquiries regarding the Bauxite SRM's 69b, 696, 697, and 698, should be directed to the Office of Standard Reference Materials, Chemistry Building, B311, National Bureau of Standards, Washington, D.C. 20234. (301) 921-2045.

George A. Uriano, Chief  
Office of Standard Reference Materials

# National Bureau of Standards Certificate of Analysis

## Standard Reference Material 697

### Bauxite (Dominican)

(In Cooperation with the American Society for Testing and Materials)

(All analyses are based on samples dried 2 hours at 140 °C)

This material is in the form of fine powder (<0.08 mm) for use in checking chemical and instrumental methods of analyses.

Constituent	Certified Value <sup>1</sup> Percent, by weight	Estimated Uncertainty <sup>2</sup>
Al <sub>2</sub> O <sub>3</sub>	45.8	0.2
Fe <sub>2</sub> O <sub>3</sub>	20.0	.2
SiO <sub>2</sub>	6.81	.07
TiO <sub>2</sub>	2.52	.05
ZrO <sub>2</sub>	0.065	.007
P <sub>2</sub> O <sub>5</sub>	.97	.06
V <sub>2</sub> O <sub>5</sub>	.063	.005
Cr <sub>2</sub> O <sub>3</sub>	.100	.005
CaO	.71	.03
MgO	.18	.02
MnO	.41	.03
ZnO	.037	.003
K <sub>2</sub> O	.062	.007
SO <sub>3</sub>	.13	.03
Loss on Ignition <sup>3</sup>	22.1	.2

<sup>1</sup> The certified value listed for a constituent is the *present best estimate* of the "true" value.

<sup>2</sup> The estimated uncertainty listed for a constituent is based on judgment and represents an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material variability for samples 1.0 g or more. (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determination of most constituents.)

<sup>3</sup> Determined by igniting to constant weight at 1050 °C.

Washington, D.C. 20234  
August 24, 1979

George A. Uriano, Chief  
Office of Standard Reference Materials

(over)

#### ADDITIONAL INFORMATION ON THE COMPOSITION

Elements other than those certified may be present in this material as indicated below. These are not certified but are given as additional information on the composition.

<u>Constituent</u>	<u>Concentration, Percent by weight</u>	<u>Constituent</u>	<u>Concentration, Percent by weight</u>
BaO	(0.015)	Co	(0.0013)
Na <sub>2</sub> O	(0.036)	Hf	(0.0014)
Ce	(0.069)	Sc	(0.0058)

The mineralogical composition of SRM 697 was determined by x-ray diffraction studies at the Geological Survey, U.S. Department of the Interior, Reston, Va., (J. W. Hosterman) to be 15% kaolinite, 50% gibbsite, 10% boehmite, 20% hematite, and 5% anatase. These results are semiquantitative (to the nearest 5%).

#### PLANNING, PREPARATION, TESTING, ANALYSIS:

The material for this SRM was mined in the Dominican Republic and was provided by the Aluminum Company of America, Alcoa Technical Center, Pittsburgh, Pa., through the courtesy of H. B. Hartman. It was processed (crushed, ground, sieved, and mixed) at the Colorado School of Mines Research Institute under a contract with the National Bureau of Standards.

Homogeneity testing was performed at NBS by J.S. Maples and T.E. Gills.

Cooperative analyses for certification were performed in the following laboratories:

Aluminum Company of America, Alcoa Center, Pa., R. C. Obbink.  
Aluminum Company of Canada, Ltd., Arvida Research Center, Arvida, Quebec, Canada, L. Girolami.  
Andrew S. McCreath & Son, Inc., Harrisburg, Pa., F. A. Pennington, Jr., R. F. Eakin, and S. L. Miller.  
General Refractories Co., U.S. Refractories Division, Research Center, Baltimore, Md., S. Banerjee.  
Geological Survey, U.S. Department of the Interior, Reston, Va., H. J. Rose, Jr., and J. W. Hosterman.  
Kaiser Aluminum and Chemical Corp., Center for Technology, Pleasanton, Calif., H. J. Seim, A. E. McLaughlin, D. F. G. Marten, A. Kermaninejad, R. C. Kinne, J. R. Skarset, J. Boruk, and U. Vogel.  
National Bureau of Standards, Washington, D.C., R. K. Bell, ASTM-NBS Assistant Research Associate.  
National-Southwire Aluminum Co., Hawesville, Ky., N. Robinson and E. Gotzy.  
Ormet Corp., Burnside, La., W. L. Brown and A. D. Lafleur.  
Reynolds Aluminum Co., Alumina Research Division, Bauxite, Ark., J. B. Ezell, Jr.  
University of Kentucky, Institute for Mining and Minerals Research, Center for Energy Research Laboratory, Lexington, Ky., T. V. Rebagay.

The overall coordination of the technical measurements leading to certification were performed under the direction of J. I. Shultz, Research Associate, ASTM-NBS Research Associate Program.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. E. Michaelis and R. Alvarez.

## NBS Standard Reference Materials

## BAUXITE SERIES

September 4, 1979

R. E. Michaelis and R. Alvarez, NBS Office of Standard Reference Materials  
and

J. I. Shultz, ASTM Research Associate

The following table gives the values for four bauxite SRM's that are available in the form of fine powder (<0.08 mm) for use in chemical and instrumental methods of analysis. They are being issued as a culmination of a major Industry-ASTM-NBS cooperative program.

SRM No. Designation	69b Arkansas	696 Surinam	697 Dominican	698 Jamaican
<u>Constituent</u>	<u>P e r c e n t   b y   W e i g h t</u>			
Al <sub>2</sub> O <sub>3</sub>	48.8	54.5	45.8	48.2
Fe <sub>2</sub> O <sub>3</sub>	7.14	8.70	20.0	19.6
SiO <sub>2</sub>	13.43	3.79	6.81	0.69
TiO <sub>2</sub>	1.90	2.64	2.52	2.38
ZrO <sub>2</sub>	0.29	0.14	0.065	0.061
P <sub>2</sub> O <sub>5</sub>	0.118	0.050	0.97	0.37
V <sub>2</sub> O <sub>5</sub>	0.028	0.072	0.063	0.064
Cr <sub>2</sub> O <sub>3</sub>	0.011	0.047	0.100	0.080
CaO	0.13	0.018	0.71	0.62
MgO	0.085	0.012	0.18	0.058
MnO	0.110	0.004	0.41	0.38
ZnO	0.0035	0.0014	0.037	0.029
BaO	(0.008) <sup>a</sup>	(0.004)	(0.015)	(0.008)
Na <sub>2</sub> O	(0.025)	(0.007)	(0.036)	(0.015)
K <sub>2</sub> O	0.068	0.009	0.062	0.010
SO <sub>3</sub>	0.63	0.21	0.13	0.22
Loss on Ign.	27.2	29.9	22.1	27.3
Ce	(0.024)	(0.0041)	(0.069)	(0.030)
Co	(0.0001)	(0.00009)	(0.0013)	(0.0045)
Hf	(0.0063)	(0.0032)	(0.0014)	(0.0015)
Sc	(0.0008)	(0.0008)	(0.0058)	(0.0051)
Total	(100.0)	(100.1)	(100.1)	(100.1)

<sup>a</sup>Values in parenthesis are *not certified*.

The value listed for a certified constituent is the *present best estimate* of the "true" value based on the results of the analytical program for certification (10-12 laboratories). The individual certificates of analysis list the "estimated uncertainties" associated with the certified values (also listed is a semiquantitative mineralogical composition (±5%) as determined by x-ray diffraction studies at the U.S. Geological Survey).

Inquiries regarding the Bauxite SRM's 69b, 696, 697, and 698, should be directed to the Office of Standard Reference Materials, Chemistry Building, B311, National Bureau of Standards, Washington, D.C. 20234. (301) 921-2045.

George A. Uriano, Chief  
Office of Standard Reference Materials

# National Bureau of Standards

## Certificate of Analysis

### Standard Reference Material 698

#### Bauxite (Jamaican)

(In Cooperation with the American Society for Testing and Materials)

(All analyses are based on samples dried 2 hours at 140 °C)

This material is in the form of fine powder (<0.08 mm) for use in checking chemical and instrumental methods of analyses.

Constituent	Certified Value <sup>1</sup> Percent, by weight	Estimated Uncertainty <sup>2</sup>
Al <sub>2</sub> O <sub>3</sub>	48.2	0.4
Fe <sub>2</sub> O <sub>3</sub>	19.6	.2
SiO <sub>2</sub>	0.69	.03
TiO <sub>2</sub>	2.38	.07
ZrO <sub>2</sub>	0.061	.009
P <sub>2</sub> O <sub>5</sub>	.37	.01
V <sub>2</sub> O <sub>5</sub>	.064	.005
Cr <sub>2</sub> O <sub>3</sub>	.080	.006
CaO	.62	.02
MgO	.058	.008
MnO	.38	.03
ZnO	.029	.002
K <sub>2</sub> O	.010	.002
SO <sub>3</sub>	.22	.03
Loss on Ignition <sup>3</sup>	27.3	.2

<sup>1</sup>The certified value listed for a constituent is the *present best estimate* of the "true" value.

<sup>2</sup>The estimated uncertainty listed for a constituent is based on judgment and represents an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material variability for samples 1.0 g or more. (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determination of most constituents.)

<sup>3</sup>Determined by igniting to constant weight at 1050 °C.

Washington, D.C. 20234  
August 24, 1979

George A. Uriano, Chief  
Office of Standard Reference Materials

(over)



#### ADDITIONAL INFORMATION ON THE COMPOSITION

Elements other than those certified may be present in this material as indicated below. These are not certified but are given as additional information on the composition.

<u>Constituent</u>	<u>Concentration, Percent by weight</u>	<u>Constituent</u>	<u>Concentration, Percent by weight</u>
BaO	(0.008)	Co	(0.0045)
Na <sub>2</sub> O	(0.015)	Hf	(0.0015)
Ce	(0.030)	Sc	(0.0051)

The mineralogical composition of SRM 698 was determined by x-ray diffraction studies at the Geological Survey, U.S. Department of the Interior, Reston, Va., (J.W. Hosterman) to be 75% gibbsite, 20% hematite, and 5% anatase. These results are semiquantitative (to the nearest 5%).

#### PLANNING, PREPARATION, TESTING, ANALYSIS:

The material for this SRM was mined in Jamaica, and was provided by the Reynolds Metals Company, Bauxite, Arkansas, through the courtesy of J. B. Ezell, Jr. It was processed (crushed, ground, sieved, and mixed) at the Colorado School of Mines Research Institute under a contract with the National Bureau of Standards.

Homogeneity testing was performed at NBS by J.S. Maples and T.E. Gills.

Cooperative analyses for certification were performed in the following laboratories:

Aluminum Company of America, Alcoa Center, Pa., R. C. Obbink.

Aluminum Company of Canada, Ltd., Arvida Research Center, Arvida, Quebec, Canada, L. Girolami.

Andrew S. McCreath & Son, Inc., Harrisburg, Pa., F. A. Pennington, Jr., R. F. Eakin, and S. L. Miller.

General Refractories Co., U.S. Refractories Division, Research Center, Baltimore, Md., S. Banerjee.

Geological Survey, U.S. Department of the Interior, Reston, Va., H. J. Rose, Jr., and J. W. Hosterman.

Kaiser Aluminum and Chemical Corp., Center for Technology, Pleasanton, Calif., H. J. Seim, A. E. McLaughlin, D. F. G. Marten, A. Kermaninejad, R. C. Kinne, J. R. Skarset, J. Boruk, and U. Vogel.

National Bureau of Standards, Washington, D.C., R. K. Bell, ASTM-NBS Assistant Research Associate.

National-Southwire Aluminum Co., Hawesville, Ky., N. Robinson and E. Gotzy.

Ormet Corp., Burnside, La., W. L. Brown and A. D. Lafleur.

Reynolds Aluminum Co., Alumina Research Division, Bauxite, Ark., J. B. Ezell, Jr.

University of Kentucky, Institute for Mining and Minerals Research, Center for Energy Research

Laboratory, Lexington, Ky., T. V. Rebagay.

The overall coordination of the technical measurements leading to certification were performed under the direction of J. I. Shultz, Research Associate, ASTM-NBS Research Associate Program.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. E. Michaelis and R. Alvarez.

## NBS Standard Reference Materials

## BAUXITE SERIES

September 4, 1979

R. E. Michaelis and R. Alvarez, NBS Office of Standard Reference Materials  
and

J. I. Shultz, ASTM Research Associate

The following table gives the values for four bauxite SRM's that are available in the form of fine powder (<0.08 mm) for use in chemical and instrumental methods of analysis. They are being issued as a culmination of a major Industry-ASTM-NBS cooperative program.

SRM No. Designation	69b Arkansas	696 Surinam	697 Dominican	698 Jamaican
Constituent	Percent by Weight			
Al <sub>2</sub> O <sub>3</sub>	48.8	54.5	45.8	48.2
Fe <sub>2</sub> O <sub>3</sub>	7.14	8.70	20.0	19.6
SiO <sub>2</sub>	13.43	3.79	6.81	0.69
TiO <sub>2</sub>	1.90	2.64	2.52	2.38
ZrO <sub>2</sub>	0.29	0.14	0.065	0.061
P <sub>2</sub> O <sub>5</sub>	0.118	0.050	0.97	0.37
V <sub>2</sub> O <sub>5</sub>	0.028	0.072	0.063	0.064
Cr <sub>2</sub> O <sub>3</sub>	0.011	0.047	0.100	0.080
CaO	0.13	0.018	0.71	0.62
MgO	0.085	0.012	0.18	0.058
MnO	0.110	0.004	0.41	0.38
ZnO	0.0035	0.0014	0.037	0.029
BaO	(0.008) <sup>a</sup>	(0.004)	(0.015)	(0.008)
Na <sub>2</sub> O	(0.025)	(0.007)	(0.036)	(0.015)
K <sub>2</sub> O	0.068	0.009	0.062	0.010
SO <sub>3</sub>	0.63	0.21	0.13	0.22
Loss on Ign.	27.2	29.9	22.1	27.3
Ce	(0.024)	(0.0041)	(0.069)	(0.030)
Co	(0.0001)	(0.00009)	(0.0013)	(0.0045)
Hf	(0.0063)	(0.0032)	(0.0014)	(0.0015)
Sc	(0.0008)	(0.0008)	(0.0058)	(0.0051)
Total	(100.0)	(100.1)	(100.1)	(100.1)

<sup>a</sup>Values in parenthesis are *not certified*.

The value listed for a certified constituent is the *present best estimate* of the "true" value based on the results of the analytical program for certification (10-12 laboratories). The individual certificates of analysis list the "estimated uncertainties" associated with the certified values (also listed is a semiquantitative mineralogical composition (±5%) as determined by x-ray diffraction studies at the U.S. Geological Survey).

Inquiries regarding the Bauxite SRM's 69b, 696, 697, and 698, should be directed to the Office of Standard Reference Materials, Chemistry Building, B311, National Bureau of Standards, Washington, D.C. 20234. (301) 921-2045.

George A. Uriano, Chief  
Office of Standard Reference Materials

# National Bureau of Standards

## Certificate of Analysis

### Standard Reference Material 1632b

#### Trace Elements in Coal

#### (Bituminous)

This Standard Reference Material (SRM) is intended for use in the calibration of apparatus and the evaluation of techniques employed in the analysis of coal or similar materials. SRM 1632b is a bituminous coal with a nominal sulfur content of 1.9%. It is in the form of a fine powder (-60 mesh).

**Certified Values of Constituent Elements:** The certified values for the constituent elements are given in Table 1. The certified values are based on measurements using proven techniques and methods. Noncertified values are given in Table 2 and are provided for information only. These values are based on measurements made using a single technique or method. While no reason exists to suspect systematic bias in the information values, no attempt was made to determine if such a bias exists that is attributable to the technique and/or method used. A list of analytical techniques and methods used for the different analyses is given in Table 3. As part of its update certification program, NBS will periodically update many of these values to certification status.

**Expiration of Certification:** The certification of SRM 1632b will be valid up to 5 years from the purchase date. Should any of the certified constituents become invalid prior to that date, purchasers will be notified by NBS.

**Use:** This material should be vacuum dried at ambient temperature for 24 hours prior to use. The certified concentrations are reported on a "dry-weight" basis, thus the concentration determined on undried samples should be adjusted for the moisture content of the sample. Typical moisture loss using the drying procedure stated above is 1.3%.

A minimum sample size of 250 mg of the dried material is required for the certified values to be valid.

This SRM should be kept in its original bottle. It should not be exposed to intense source of radiation, including ultraviolet lamps or sunlight.

The statistical analysis of the certification data was performed by R. C. Paule of the National Measurement Laboratory.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. E. Gills.

Gaithersburg, MD 20899  
June 20, 1985

Stanley D. Rasberry, Chief  
Office of Standard Reference Materials

(over)

Source and Preparation of Material: The coal for this SRM was obtained from the Humphrey No. 7 mine and coal preparation plant of the Consolidation Coal Company, Christopher Coal Company Division, Osage, West Virginia. This mine produces bituminous coal with a sulfur content of 1.8-1.9 percent (dry basis). This coal was obtained from an underground mine that recovers coal from the Pittsburgh seam, which is considered the single most valuable and extensive coal seam in the United States.

Approximately 900 kg of the coal for SRM 1632b was oven dried prior to processing, in accordance with procedures outlined in ASTM D2013. The coal was reduced in size to -60 mesh and sieved prior to blending. The coal was then blended in a stainless steel cone blender (approximate capacity 0.85 cubic meter). After blending the coal was packaged in polyethylene-lined aluminum cans and was subsequently repackaged in fifty gram units.

#### Analysis

Major, Minor, and Trace Constituents: In general, the major, minor, and trace constituents were certified using two or more independent methods of analysis or two or more different laboratories. For those constituents that were determined using a single method, technique, or laboratory, the values are given for information only. (See Table 3).

Calorific Value: The calorific value was determined using measurements made in an isoperibol calorimeter, an isothermal calorimeter, and an adiabatic calorimeter at two different laboratories.

Moisture, Ash, and Volatile Matter: The moisture, ash, and volatile matter values were determined on measurements made using the standard ASTM methods, D3173, D3174, and D3175, respectively. In addition, commercial instruments commonly used for the determination of the parameters provided additional values.

Table 1. Certified Values of Constituent Elements

<u>Major Constituents</u>		<u>Minor Constituents</u>	
<u>Elements</u>	<u>Content</u> <u>Wt. Percent</u>	<u>Elements</u>	<u>Content</u> <u>Wt. Percent</u>
Carbon (Total)	78.11 ± 0.37 <sup>a</sup>	Aluminum	0.855 ± 0.019
Hydrogen	5.07 ± 0.06	Calcium	0.204 ± 0.006
Nitrogen	1.56 ± 0.07	Iron	0.759 ± 0.045
Sulfur	1.89 ± 0.06	Magnesium	0.0383 ± 0.0008
Volatile matter	35.4 ± 1.1	Potassium	0.0748 ± 0.0028
		Sodium	0.0515 ± 0.0011
		Titanium	0.0454 ± 0.0017

<u>Trace Constituents</u>			
<u>Element</u>	<u>Content</u> <u>µg/g</u>	<u>Element</u>	<u>Content</u> <u>µg/g</u>
Arsenic	3.72 ± 0.09	Manganese	12.4 ± 1.0
Barium	67.5 ± 2.1	Nickel	6.10 ± 0.27
Cadmium	0.0573 ± 0.0027	Rubidium	5.05 ± 0.11
Cobalt	2.29 ± 0.17	Selenium	1.29 ± 0.11
Copper	6.28 ± 0.30	Thorium	1.342 ± 0.036
Lead	3.67 ± 0.26	Uranium	0.436 ± 0.012
		Zinc	11.89 ± 0.78

Calorific Value<sup>b,c</sup>

14005 ± 35 Btu/lb (32.57 ± 0.08 MJ kg<sup>-1</sup>)

Ash, wt. %

6.79 ± 0.16

<sup>a</sup> The listed ± uncertainties for carbon, hydrogen, volatile matter, and calorific value are two standard deviations of the certified value. The listed ± uncertainties for all other constituents are two standard deviations for the certified values and include an allowance for minor sample heterogeneity. The observed sample variability was generally less than two percent of the constituent value.

<sup>b</sup> The calorific value (MJ kg<sup>-1</sup>) may decrease upon aging or normal oxidation of the coals. NBS will continue to monitor this value and report any substantive change in the certified calorific value to the purchaser. The reference date for the calorific value is May 1985.

<sup>c</sup> The calorific value is determined as HHV2 (Higher Heating Value-Moisture Free).

Table 2. Noncertified Values for Constituent Elements

<u>Trace Constituents</u>			
<u>Element</u>	<u>Content</u> <u>µg/g</u>	<u>Element</u>	<u>Content</u> <u>µg/g</u>
Antimony	(0.24)	Lithium	(10)
Bromine	(17)	Molybdenum	(0.9)
Cerium	(9)	Samarium	(0.87)
Cesium	(0.44)	Scandium	(1.9)
Chlorine	(1260)	Silicon, wt %	(1.4)
Chromium	(11)	Strontium	(102)
Europium	(0.17)	Tungsten	(0.48)
Hafnium	(0.43)	Vanadium	(14)
Lanthanum	(5.1)		

Table 3. Analytical Techniques and Methods Used for the Characterization of SRM 1632b

Method/ Element	A	B	C	D	E	F	G	H	I	J	K	L	M
Al			*	*								*	
As			*		*								
Ash Content							* 2		*				
Ba			*										
Br			*										
C (Total)							* 5	*	*		*		
Ca		*	*	*								*	
Cal Val									*	*			
Cd	*	*											
Ce			*										
Cl			*										
Co			*	*									
Cr			*										*
Cs			*										
Cu					*								*
Eu			*										
Fe	*		*									*	
H							* 5	*	*				
Hf			*										
K		*	*	*								*	
La			*										
Li				*									
Mg	*	*	*										
Mn			*	*									
Mo			*										
N							* 6						
Na			*	*									
Ni	*												*
Pb	*	*											
Rb		*	*	*									
S						*	* 4		*			*	
Sb			*										
Sc			*										
Se			*		*								
Si			*									*	
Sm			*										
Sr			*										
Th		*	*										
Ti			*	*								*	*
U		*	*										
V			*										*
Volatile Matter							* 3		*				
W			*										
Zn	*	*	*										

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SRM 1632b

#### Analytical Methods

- A. Atomic absorption spectrometry
- B. Isotope dilution mass spectrometry
- C. Instrumental neutron activation analysis
- D. Flame emission spectrometry
- E. Flameless atomic absorption spectrometry
- F. Ion chromatography
- G. ASTM Methods: (1)D3173, (2)D3174, (3)D3175, (4)D3177, (5)D3178, (6)D3179
- H. Combustion coulometry
- I. Commercial coal analyzers: moisture, ash, sulfur, Btu, volatile matter, carbon, hydrogen, nitrogen
- J. Commercial calorimeter
- K. Gas chromatography
- L. X-ray fluorescence
- M. Inductively coupled plasma emission spectrometry

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# National Bureau of Standards

## Certificate of Analysis

### Standard Reference Material 1633a

#### Trace Elements in Coal Fly Ash

This Standard Reference Material is intended for use in the calibration of apparatus and methods used in analyses of coal fly ash and other materials with similar matrices for trace elements. This material should be dried to a constant weight before using. Recommended procedures for drying are: (1) drying for 24 hours at ambient temperature using a cold trap at or below  $-50^{\circ}\text{C}$  and a pressure not greater than 30 Pa (0.2 mm Hg); (2) drying in a desiccator over  $\text{P}_2\text{O}_5$  or  $\text{Mg}(\text{ClO}_4)_2$ . When not in use, the material should be kept in a tightly sealed bottle. Long term ( $>3$  years) stability of this SRM has not been rigorously established. NBS will continue to monitor this material and any substantive change will be reported to purchasers.

The certified values given below are based on at least a 250-mg sample of the dried material, the minimum amount that should be used for analysis.

Element <sup>1</sup>	Content <sup>2</sup>	Element	Content
	<u>ppm</u>		<u>μg/g</u>
Calcium <sup>a,b,e</sup>	1.11 ± 0.01	Mercury <sup>a,c</sup>	0.16 ± 0.01
Iron <sup>a,b,c</sup>	9.40 ± 0.10	Nickel <sup>b,d,e</sup>	127 ± 4
Potassium <sup>a,b,e</sup>	1.88 ± 0.06	Lead <sup>b,d,e</sup>	72.4 ± 0.4
Magnesium <sup>a,b</sup>	0.455 ± 0.010	Rubidium <sup>a,b,c,e</sup>	131 ± 2
Sodium <sup>a,c</sup>	0.17 ± 0.01	Selenium <sup>a,c,g</sup>	10.3 ± 0.6
Silicon <sup>a,h</sup>	22.8 ± 0.8	Strontium <sup>a,e,f</sup>	830 ± 30
	<u>μg/g</u>	Thorium <sup>b,c</sup>	24.7 ± 0.3
Arsenic <sup>a,c</sup>	145 ± 15	Thallium <sup>b,g</sup>	5.7 ± 0.2
Cadmium <sup>b,c,d,g</sup>	1.0 ± 0.15	Uranium <sup>b</sup>	10.2 ± 0.1
Chromium <sup>a,b,c</sup>	196 ± 6	Zinc <sup>a,b,d,e,f</sup>	220 ± 10
Copper <sup>a,b,c</sup>	118 ± 3		

#### 1. Methods of Analysis:

<sup>a</sup>Atomic Absorption Spectrophotometry or Flame Emission Spectrometry

<sup>b</sup>Isotope Dilution Mass Spectrometry

<sup>c</sup>Neutron Activation

<sup>d</sup>Polarography

<sup>e</sup>X-ray Fluorescence Spectrometry

<sup>f</sup>Inductively Coupled Plasma Emission Spectrometry

<sup>g</sup>Isotope Dilution Spark Source Mass Spectrometry

<sup>h</sup>Gravimetry

2. The estimated uncertainty is based on judgment and represents an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material variability for samples of 250-mg or more. (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determination of most constituents.)

Washington, D.C. 20234  
 April 18, 1979

George A. Urano, Chief  
 Office of Standard Reference Materials

(over)



The overall direction and coordination of the analytical measurements leading to certification were performed in the Center for Analytical Chemistry under the chairmanship of L. A. Machlan.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by W. P. Reed.

#### PREPARATION, TESTING, AND ANALYSIS

This fly ash material was supplied by a coal fired power plant and is a product of Pennsylvania and West Virginia coals. It was selected as a typical fly ash and is not intended as a fly ash from a specific coal or combustion process. The material was sieved through a # 170 sieve and blended for 2 hours in a Vee blender. The material was then removed and placed in a series of bulk containers from which specific samples were taken.

Twelve bottles were selected for homogeneity tests. These samples were analyzed for cobalt, chromium, europium, iron, scandium, and thorium by nondestructive neutron activation analysis. The observed standard deviations for both 50 and 250 mg samples were consistent with counting statistics indicating that the fly ash is homogeneous within  $\pm 5\%$  (relative) based on these elements. The homogeneity analyses were performed in the NBS Center for Analytical Chemistry by R. R. Greenburg and J. S. Maples. Analyses for the various elements were made in the NBS Center for Analytical Chemistry by the following analysts: J. R. Baldwin, T. J. Brady, E. R. Deardorff, M. G. Dias, L. P. Dunstan, M. S. Epstein, E. L. Garner, T. E. Gills, C. A. Grabnegger, J. W. Gramlich, R. R. Greenberg, S. Hanamura, S. H. Harrison, E. F. Heald, H. M. Kingston, E. C. Kuehner, L. A. Machlan, E. J. Maienthal, J. S. Maples, J. D. Messman, L. J. Moore, P. J. Paulsen, P. A. Pella, T. C. Rains, K. J. R. Rosman, T. A. Rush, P. A. Sleeth, and R. L. Waters, Jr.

The following values are not certified because they are based on a non-reference method, or were not determined by two or more independent methods. They are included for information only.

Element	Content	Element	Content
	<u>%</u>		<u>µg/g</u>
Aluminum	14	Europium	4
Barium	0.15	Gallium	58
Titanium	0.8	Hafnium	7.6
	<u>µg/g</u>	Manganese	190
Beryllium	12	Molybdenum	29
Cerium	180	Antimony	7
Cobalt	46	Scandium	40
Cesium	11	Vanadium	300

# National Bureau of Standards

## Certificate of Analysis

### Standard Reference Material 1635

#### Trace Elements in Coal (Subbituminous)

This Standard Reference Material is intended for use in the calibration of apparatus and the evaluation of techniques employed in the trace element analysis of coal and similar materials. The material should be dried without heat to constant weight before use.

The recommended procedures for drying are either vacuum drying at ambient temperature for 24 hours, or freeze drying in which the drying chamber is kept at room temperature. The moisture content of this material is approximately 20%. Because of this moisture level, it is recommended that small individual samples be dried immediately before use. Drying of large samples may result in a violent discharge of water vapor and resultant loss of sample. When not in use, the material should be kept in a tightly sealed bottle and stored in a cool, dark place. Long-term (>1 year) stability of this SRM has not been rigorously established. NBS will continue to monitor this material and any substantive change will be reported to purchasers.

The certified values given below are based on at least a 250-mg sample of the dried material, the minimum amount that should be used for analysis.

Element <sup>1</sup>	Content, $\mu\text{g/g}^2$	Element <sup>1</sup>	Content, $\mu\text{g/g}^2$
Arsenic <sup>a,b</sup>	$0.42 \pm 0.15$	Thorium <sup>c,e</sup>	$0.62 \pm 0.04$
Cadmium <sup>c,d,e</sup>	$0.03 \pm 0.01$	Uranium <sup>c</sup>	$0.24 \pm 0.02$
Chromium <sup>c,e</sup>	$2.5 \pm 0.3$	Vanadium <sup>c,g</sup>	$5.2 \pm 0.5$
Copper <sup>a,c,e</sup>	$3.6 \pm 0.3$	Zinc <sup>c,d</sup>	$4.7 \pm 0.5$
Lead <sup>c,d</sup>	$1.9 \pm 0.2$		
Manganese <sup>a,c</sup>	$21.4 \pm 1.5$	Element <sup>1</sup>	Wt. % <sup>2</sup>
Nickel <sup>c,d</sup>	$1.74 \pm 0.10$	Iron <sup>c,d,e,i</sup>	$0.239 \pm 0.005$
Selenium <sup>a,e</sup>	$0.9 \pm 0.3$	Sulfur <sup>f,h</sup>	$0.33 \pm 0.03$

1. Methods of Analysis:

- |                                       |                                |
|---------------------------------------|--------------------------------|
| a. Atomic Absorption Spectrometry     | e. Neutron Activation          |
| b. Photon Activation                  | f. Spectrophotometry           |
| c. Isotope Dilution Mass Spectrometry | g. Flame Emission Spectrometry |
| d. Polarography                       | h. Gravimetry                  |

2. The estimated uncertainty is based on judgment and represents an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material variability for samples of 250-mg or more. (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determination of most constituents.)

The overall direction and coordination of the analytical measurements leading to this certificate were performed in the Analytical Chemistry Division under the chairmanship of L. J. Moore.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by W. P. Reed.

Washington, D.C. 20234  
August 22, 1979  
(Revision of Certificate  
dated 1-23-78)

George A. Uriano  
Office of Standard Reference Materials

(over)

#### PREPARATION, TESTING, and ANALYSIS

This material was prepared from one lot of subbituminous coal from the Eagle Mine of The Imperial Coal Company, Erie, Colorado. The material was ground and sieved thru a No. 65 (230  $\mu\text{m}$ ) sieve by the Colorado School of Mines Research Institute. The material was then blended in a V-type blender.

Samples for homogeneity testing were taken from the top, middle, and bottom of three bulk containers of coal, and analyzed by neutron activation analysis for sodium, scandium, chromium, iron, cobalt, lanthanum, cerium, and thorium. Replicate analyses of 250-mg samples indicated a homogeneity for these elements of  $\pm 2.5\%$  (relative) except for chromium, which was homogeneous within counting statistics of  $\pm 6\%$ . The homogeneity measurements were performed in the NBS Analytical Chemistry Division by R. R. Greenberg. Certification analyses for the various elements were made in the NBS Analytical Chemistry Division by T. J. Brady, B. I. Diamondstone, L. P. Dunstan, M. S. Epstein, M. Gallorini, E. L. Garner, T. E. Gills, J. W. Gramlich, R. R. Greenberg, S. H. Harrison, G. M. Hyde, G. J. Lutz, L. A. Machlan, E. J. Maienthal, J. D. Messman, T. J. Murphy, and T. C. Rains.

The following values are *not certified* because they were based on a non-reference method, or were not determined by two or more independent methods. They are included for information only.

<u>Element</u>	<u>Content</u> <u>(<math>\mu\text{g/g}</math>)</u>
Antimony	(0.14)
Cerium	(3.6)
Cobalt	(0.65)
Europium	(0.06)
Gallium	(1.05)
Hafnium	(0.29)
Scandium	(0.63)
	<u>(wt. %)</u>
Aluminum	(0.32)
Sodium	(0.24)
Titanium	(0.02)

# National Bureau of Standards

## Certificate of Analysis

### Standard Reference Materials

2682, 2683, 2684 and 2685

#### Sulfur in Coal

These Standard Reference Materials (SRM's) are intended primarily for use as analytical standards for the determination of sulfur in coal. In addition to sulfur they are certified for their calorific value ( $\text{MJ} \cdot \text{kg}^{-1}$ ) and ash content. SRM's 2682-2685 each consists of a 50-g bottle of a different coal composition. Each material was ground to pass a 60-mesh sieve and homogenized. The certification of the materials for sulfur is based on at least a 250-mg sample of the dried material, the minimum amount that should be used for analysis (see drying instructions). The calorific values were determined by procedures recommended in standard ASTM methods (see references in Table 1). The certification data for the four different coals are given in Table 1 along with methods used for certification. Noncertified values for major and minor elements are given in Table 2. These values are provided for information only.

Notice to Users: These SRM's are sold individually rather than in sets; however, only one Certificate of Analysis is provided. Therefore, the user must be careful to use the data specific to the SRM being used.

The uncertainty of a certified value is expressed as two times the standard error and includes observed variability within and between measurement methods and any observed material heterogeneity. For the certified heating values the uncertainty also includes additional allowances for possible limited sample degradation due to aging or normal oxidation.

Certification analyses were performed by W.R. Kelly, W.F. Koch, P.J. Paulsen, and J.W. Stolz of the Inorganic Analytical Research Division and J.C. Colbert and D.R. Kirklin of the Chemical Thermodynamics Division.

Analyses for supplemental information were performed in the Inorganic Analytical Research Division by R. Fleming, R. Greenberg, and R.M. Lindstrom.

The statistical analysis of the certification data was performed by R.C. Paule of the National Measurement Laboratory.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of E.L. Garner, Chief of the Inorganic Analytical Research Division.

The technical and support aspects involved in the preparation, certification, and issuance of these Standard Reference Materials were coordinated through the Office of Standard Reference Materials by T.E. Gills.

February 7, 1983  
(Revision of Certificate  
dated 12-14-82)

(over) George A. Uriano, Chief  
Office of Standard Reference Materials

Table 1  
Certified Values for SRM's 2682, 2683, 2684, and 2685

SRM No.	Coal Type	Sulfur <sup>1,2,3</sup> Wt. %	Furnace <sup>4</sup> Ash Wt. %	HHV2 <sup>5,6</sup> MJ·Kg <sup>-1</sup> (BTU·lb <sup>-1</sup> )
2682	Sub-bituminous	0.47 ± 0.03	6.37 ± 0.18	27.45 ± 0.56 (11800 ± 240)
2683	Bituminous	1.85 ± 0.06	6.85 ± 0.02	32.70 ± 0.14 (14060 ± 60)
2684	Bituminous	3.00 ± 0.13	11.09 ± 0.18	29.68 ± 0.47 (12760 ± 200)
2685	Bituminous	4.62 ± 0.18	16.53 ± 0.15	28.15 ± 0.42 (12100 ± 180)

<sup>1</sup>ASTM D3177 Standard Test Method for Total Sulfur in the Analysis Sample of Coal and Coke

<sup>2</sup>Ion Chromatography with Bomb Combustion

<sup>3</sup>Thermal Ionization Mass Spectrometry, Sealed Glass Tube Digestion

<sup>4</sup>ASTM D3174, Standard Test Method for Ash in the Analysis Sample of Coal and Coke

<sup>5</sup>ASTM D2015 Standard Test Method for Gross Calorific Value of Solid Fuel by the Adiabatic Bomb Calorimeter

<sup>6</sup>ASTM D3189 Standard Test Method for Calculating Coal and Coke Analyses from As-Determined to Different Bases  
HHV2=(Higher Heating Value - Moisture Free)

Note: The calorific values (MJ·Kg<sup>-1</sup>) may decrease upon the aging or normal oxidation of the coals. NBS will continue to monitor these calorific values and report any substantive change to the purchaser. The uncertainty of the heating value includes an additional allowance of 0.12 MJ·Kg<sup>-1</sup> for possible sample degradation. The reference date for the calorific data is October 1982.

#### PREPARATION AND TESTING

Approximately one ton of coal was obtained from each of four different coal mine locations. All coals were oven dried prior to processing in accordance with procedures outlined in ASTM D2013. At least 1000 pounds of each of the four coals were reduced in size to -60 mesh and screened prior to blending. Each of the -60 mesh coals was blended in a stainless steel cone blender (approximate capacity 0.85 cubic meter). The coals were then packaged into individual 50-g bottled units. Homogeneity testing was done on the bulk materials and 50-g bottled units. The homogeneity analyses were performed using x-ray fluorescence analysis. Replicate analyses indicated the material variability for sulfur to be within ± 2% (relative) for all four SRM's.

The homogeneity studies were performed by T.E. Gills and M. Watson of the Office of Standard Reference Materials and P.A. Pella of the Gas and Particulate Science Division.

#### ANALYSIS

Sulfur: The certified sulfur content is based upon the results of 3 independent methods of analysis: ion chromatography, gravimetry, and thermal ionization mass spectrometry. Agreement with the certified values was found using 2 additional independent techniques, prompt-gamma activation analysis and a combustion IR technique.

Calorific Value ( $\text{MJ}\cdot\text{Kg}^{-1}$ ) and Ash Content: The certified values for the calorific values and ash contents were determined using measurements made in an adiabatic bomb calorimeter of the type used in commercial laboratories. This calorimeter is capable of reproducing determinations on benzoic acid to a precision of 0.07% (relative). This statement of precision was arrived at by averaging 5 calibrations made on the calorimeter using a benzoic acid standard that is traceable to the NBS SRM 39i, Benzoic Acid.

Major and Minor Elements: Analyses for major and minor elements were performed by thermal neutron activation analysis and neutron capture gamma-ray activation analysis. These values are not certified but are to be used for information only.

#### STABILITY

The long-term physical and chemical stability of these SRM's has not been rigorously established. However, NBS recommends that the material be stored in the tightly sealed bottle away from sunlight and intense sources of radiation. NBS will continue to monitor these materials and any substantive change in their certification will be reported to the purchaser.

#### INSTRUCTIONS FOR DRYING

The certification of sulfur in these SRM's is based upon a properly dried sample. The recommended procedures for drying are vacuum drying at ambient temperature for 24 hours or oven drying for 24 hours at 105 °C. Typical moisture loss using the recommended methods for drying are the following: SRM 2682, 1.8%; SRM 2683, 1.4%; SRM 2684, 3.6%; and SRM 2685, 1.8%.

#### SUPPLEMENTAL INFORMATION

The values listed below are based on measurements made using a single method or technique and are given for *information only*. While no reason exists to suspect systematic bias in these numbers, no attempt was made to determine if such bias attributable to the methods exists.

The analyses of SRM's 2682-2685 for major and minor elements were made using NBS SRM's 1632a and 1635, Trace Elements in Coal, as controls.

Table 2  
Inorganic Constituents in SRM's 2682, 2683, 2684, and 2685  
Mean Concentrations ( $\mu\text{g/g}$ ) Unless Noted

Element/SRM	2682	2683	2684	2685
Al %	0.46	0.86	1.1	1.7
As	1.0	3.6	3.9	12
B	39	67	114	109
Ba	382	71	41	105
Br	3.7	17	11	5.6
C %	75	79	68	66
Ca %	1.1	0.20	0.44	0.52
Ce	10	9	12	18
Co	1.7	2.2	3.9	4.6
Cr	15	11	17	22
Cs	<0.1	0.4	1.2	1.3
Eu	0.17	0.18	0.23	0.36
Fe %	0.24	0.76	1.5	2.9
H %	4.7	5.0	4.8	4.6
Hf	0.60	0.42	0.57	0.91
K %	0.01	0.08	0.20	0.26
La	5.2	5.1	6.7	10
Mg %	0.2	0.05	0.08	0.1
Mn	26	13	36	41
N %	0.8	1.6	1.6	1.1
Na %	0.10	0.05	0.03	0.08
Rb	<2	5.3	15	17
Sb	0.19	0.28	0.35	0.36
Sc	1.5	1.9	2.7	3.7
Se	0.91	1.2	1.9	1.9
Sm	0.78	0.86	1.1	1.7
Th	1.5	1.4	2.0	2.7
Ti %	0.05	0.04	0.06	0.09
U	0.52	0.42	0.90	0.95
V	15	14	22	31
W	1.8	0.48	0.56	1.2
Zn	8.6	9.5	110	17









# **NBS** *Technical Publications*

## *Periodical*

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